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**1994**

**PERFORMANCE REPORT**

**WATER QUALITY ANALYSES SECTION**

**OCTOBER 1995**

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380  
P47  
1994  
MOE



**Ministry of  
Environment  
and Energy**

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**PERFORMANCE REPORT**

**WATER QUALITY ANALYSES SECTION**

Susan Janhurst (ed.)

Laboratory Services Branch  
Ontario Ministry of the Environment and Energy

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## INTRODUCTION

The Water Quality Analyses Section (WQAS) is part of the Ministry of the Environment's and Energy Laboratory Services Branch. The WQAS provides expertise in inorganic chemistry and handles the largest number of tests in the branch where technologists analyze a broad spectrum of environmental sample types including: ground water, surface water, drinking water, precipitation, sewage, industrial waste, landfill leachate, soil and soil extract.

This report provides a brief outline of the analytical quality control (QC) program associated with sample analysis and, summarizes the 1994 performance data for each test. WQAS strives to maintain a high standard of analytical performance through its quality assurance program and QC is an integral part of the process.

## ACKNOWLEDGEMENTS

The editor would like to thank; the technical staff in the Water Quality Analyses Section for their assistance in Quality Control data collection; Priscilla Wong for conducting statistical data summaries and updating the report to reflect the changes of the new Laboratory Information Management System; the supervisors and manager for their reviews of this document.

## TABLE OF CONTENTS

|   |    |
|---|----|
| <b>PART 1.0 Performance Report Format</b> ..... | 1  |
| <b>PART 2.0 Chemistry</b> .....                 | 4  |
| 2.1 Quality Control Program, Chemistry .....    | 5  |
| 2.3 Performance Summaries, Chemistry .....      | 8  |
| <b>Acidity, Gran</b>                            |    |
| Titration (E3248A) .....                        | 9  |
| <b>Acidity, Total Fixed Endpoint</b>            |    |
| Titration (E3248A) .....                        | 12 |
| <b>Alkalinity, Gran</b>                         |    |
| Dorset (E3042A) .....                           | 15 |
| Titration (E3289A) .....                        | 18 |
| <b>Alkalinity, Total Fixed Endpoint 4.5</b>     |    |
| Dorset (E3042A) .....                           | 21 |
| Titration (E3218A) .....                        | 24 |
| Titration (E3228A) .....                        | 27 |
| Titration (E3289A) .....                        | 30 |
| <b>Alkalinity Total Fixed Endpoint 3.8</b>      |    |
| Dorset (E3042A) .....                           | 33 |
| <b>Aluminum, Reactive Species</b>               |    |
| Dorset (E3020A,E3256A) .....                    | 36 |
| <b>Aluminum, Total</b>                          |    |
| Dorset (E3300A) .....                           | 42 |
| <b>Cadmium, Total</b>                           |    |
| Dorset (E3376A) .....                           | 45 |
| <b>Calcium</b>                                  |    |
| Atomic Absorption (E3146A) .....                | 48 |
| Atomic Absorption (E3171A) .....                | 51 |
| Atomic Absorption (E3217A) .....                | 54 |
| Dorset (E3249A) .....                           | 57 |
| <b>Carbon, Dissolved Inorganic</b>              |    |
| Dorset (E3028A) .....                           | 61 |
| Colourimetry (E3370A) .....                     | 63 |
| <b>Carbon, Dissolved Organic</b>                |    |
| Colourimetry (E3370A) .....                     | 68 |
| <b>Carbon, Total Organic</b>                    |    |
| MISA (E3247B) .....                             | 71 |
| <b>Chloride</b>                                 |    |
| Colourimetry (E3016A) .....                     | 74 |
| Dorset (E3147A) .....                           | 77 |
| Ion Chromatography (E3148A) .....               | 80 |
| Ion Chromatography (E3372A) .....               | 83 |
| <b>Chlorine, Total Residual</b>                 |    |
| MISA (E3809A) .....                             | 86 |
| <b>Chlorophyll "a"</b>                          |    |
| Colourimetry (E3169A) .....                     | 89 |
| <b>Chlorophyll "a"acidified</b>                 |    |
| Colourimetry (E3169A) .....                     | 92 |

## TABLE OF CONTENTS CONT'D

|  |     |
|--|-----|
| <b>Chlorophyll "b"</b>                 |     |
| Colourimetry (E3169A) .....            | 94  |
| <b>Colour, True</b>                    |     |
| Dorset (E3025A) .....                  | 96  |
| Colourimetry (E3219A) .....            | 99  |
| <b>Conductivity</b>                    |     |
| Dorset (E3024B) .....                  | 102 |
| Ion Chromatography (E3177A) .....      | 105 |
| Titration (E3218A) .....               | 108 |
| Titration (E3228A) .....               | 111 |
| Titration (E3289A) .....               | 114 |
| <b>Copper, Total</b>                   |     |
| Dorset (E3376A) .....                  | 117 |
| <b>Cyanide, Free</b>                   |     |
| Colourimetry (E3014A) .....            | 120 |
| <b>Cyanide, Total</b>                  |     |
| Colourimetry (E3015A) .....            | 123 |
| <b>Fluoride</b>                        |     |
| Dorset (E3041A) .....                  | 126 |
| Colourimetry (E3369A) .....            | 129 |
| <b>Hardness</b>                        |     |
| Atomic Absorption (E3171A) .....       | 132 |
| Atomic Absorption (E3217A) .....       | 133 |
| Dorset (E3249A) .....                  | 134 |
| <b>Iron, Total</b>                     |     |
| Dorset (E3303B) .....                  | 135 |
| <b>Lead, Total</b>                     |     |
| Dorset (E3376A) .....                  | 138 |
| <b>Magnesium</b>                       |     |
| Atomic Absorption (E3146A) .....       | 141 |
| Atomic Absorption (E3171A) .....       | 144 |
| Atomic Absorption (E3217A) .....       | 147 |
| Dorset (E3249A) .....                  | 150 |
| <b>Manganese, Total</b>                |     |
| Dorset (E3303B) .....                  | 153 |
| <b>Nitrogen, Ammonia plus Ammonium</b> |     |
| Colourimetry (E3149A) (mg/L) .....     | 156 |
| Colourimetry (E3364A) .....            | 159 |
| Colourimetry (E3366A) .....            | 162 |
| Dorset (E3374A) .....                  | 165 |
| <b>Nitrogen, Nitrate</b>               |     |
| Ion Chromatography (E3148A) .....      | 168 |
| Ion Chromatography (E3372A) .....      | 172 |

## TABLE OF CONTENTS CONT'D

|   |     |
|---|-----|
| <b>Nitrogen, Nitrate plus Nitrite</b>       |     |
| Colourimetry (E3364A) .....                 | 175 |
| Colourimetry (E3366A) .....                 | 178 |
| Colourimetry (E3369A) .....                 | 181 |
| Dorset (E3374A) .....                       | 184 |
| <b>Nitrogen, Nitrite</b>                    |     |
| Colourimetry (E3364A) .....                 | 187 |
| Colourimetry (E3366A) .....                 | 190 |
| <b>Nitrogen, Total Kjeldahl</b>             |     |
| Colourimetry (E3116A) .....                 | 193 |
| Colourimetry (E3118A) .....                 | 196 |
| Colourimetry (E3367A) .....                 | 199 |
| Colourimetry (E3368A) .....                 | 202 |
| <b>Oxygen Demand, Biochemical</b>           |     |
| BOD (E3182A) .....                          | 205 |
| <b>Oxygen Demand, Chemical</b>              |     |
| Colourimetry (E3170A) .....                 | 209 |
| Colourimetry (E3246A) .....                 | 212 |
| <b>pH</b>                                   |     |
| Dorset (E3042A) .....                       | 215 |
| Titration (E3218A) .....                    | 218 |
| Titration (E3228A) .....                    | 221 |
| Titration (E3248A) .....                    | 224 |
| Titration (E3289A) .....                    | 227 |
| <b>Phenolics, Reactive</b>                  |     |
| Colourimetry (E3179A) .....                 | 230 |
| <b>Phosphorus, Reactive ortho-Phosphate</b> |     |
| Colourimetry (E3364A) .....                 | 233 |
| Colourimetry (E3366A) .....                 | 236 |
| <b>Phosphorus, Total</b>                    |     |
| Dorset (E3036A) .....                       | 239 |
| <b>Phosphorus, Total</b>                    |     |
| Colourimetry (E3116A) .....                 | 242 |
| Colourimetry (E3118A) .....                 | 245 |
| Colourimetry (E3367A) .....                 | 248 |
| Colourimetry (E3368A) .....                 | 251 |
| <b>Potassium</b>                            |     |
| Atomic Absorption (E3146A) .....            | 254 |
| Atomic Absorption (E3171A) .....            | 257 |
| Atomic Absorption (E3217A) .....            | 260 |
| Dorset (E3249A) .....                       | 263 |
| <b>Silicon, Reactive Silicates</b>          |     |
| Colourimetry (E3370A) .....                 | 266 |

## TABLE OF CONTENTS CONT'D

|   |     |
|---|-----|
| <b>Sodium</b>   |     |
| Atomic Absorption (E3146A) .....  | 269 |
| Atomic Absorption (E3171A) .....  | 272 |
| Atomic Absorption (E3217A) .....  | 275 |
| Dorset (E3249A) .....   | 278 |
| <b>Solids, Dissolved</b>  |     |
| Solids (E3188B) .....   | 281 |
| River Solids (E3365A) .....   | 284 |
| <b>Solids, Suspended</b>  |     |
| Solids (E3188B) .....   | 287 |
| River Solids (E3365A) .....   | 290 |
| <b>Solids, Suspended Ignited (Particulate Ash and Particulate Loss on Ignition)</b> |     |
| Solids (E3188B) .....   | 293 |
| <b>Solids, Total</b>  |     |
| Solids (E3188B) .....   | 297 |
| Solids (E3365A) .....   | 300 |
| <b>Solids, Total Ignited ( Ash and Loss on Ignition)</b>                            |     |
| Solids (E3188B) .....   | 303 |
| <b>Sulphate</b>   |     |
| Dorset (E3147A) .....   | 307 |
| Ion Chromatography (E3148A) .....   | 310 |
| Ion Chromatography (E3172A) .....   | 315 |
| Ion Chromatography (E3372A) .....   | 318 |
| <b>Turbidity</b>  |     |
| Colourimetry (E3311A) .....   | 321 |
| <b>Zinc, Total</b>  |     |
| Dorset (E3376A) .....   | 325 |
| <b><u>Bibliography</u></b> .....  | 328 |
| <b>Abbreviations</b> .....  | 329 |
| <b>Appendix A (W and T Reporting)</b> .....   | 331 |
| <b>Appendix B (Table of Tests)</b> .....  | 332 |



## 1.0 PERFORMANCE REPORT FORMAT

The performance report is divided into two parts. Part One provides details on the report structure and, Part Two indicates the annual performance summaries of the quality control data for WQAS chemistry tests with the exception of those parameters where no data or less than three pieces of data exist for 1994. The summary usually consists of three pages: the test description page, the performance data summary page, and the quality control graphics page (Graphs are not presented where less than ten pieces of data have been collected).

The performance report is organized first, alphabetically according to test name (eg. Total Organic Carbon is filed under the heading "Carbon, Total Organic") and second, by the method reference number. In September of 1994, with the exception of Dorset Laboratory, the Toronto laboratory switched from the Laboratory Information System (LIS) to the Laboratory Information Management System (LIMS). Detailed information concerning each of these pages is outlined next.

### 1.1 TEST DESCRIPTION PAGE

#### TITLE:

The name of the test parameter.

#### IDENTIFICATION:

Laboratory:

Location where the test is performed.

LIS Test Name Code:

LIS code for analysis request.

LIMS Product Code:

LIMS code for analysis request.

Work Station Code:

LIS code for sample routing to the work station.

Method Reference No:

A number assigned by the Quality Management Office to an analytical test method eg.(E3228A). E3 denotes a Central Regional Lab test method. The subsequent three numbers are issued sequentially per method. A letter at the end denotes revision status.

Sample Type/Matrix:

The various sample types that can be routed to the work station.

Method Introduced:

Date that the method was implemented at the laboratory.

Units:

Unit of measurement in which the results are reported.

Unit Code:

LIS code for the unit of measurement in which the results are reported.

Supervisor:

Name of supervisor responsible for the designated laboratory.

SAMPLING:

The type of container and preservative (if applicable) that is used and minimum volume of sample that is usually required (7). Any sample preparation that is normally performed in the field, is also indicated.

SAMPLE PREPARATION:

Sample preparation techniques which are usually performed at the laboratory before analysis.

ANALYTICAL PROCEDURE:

Analytical method used to determine the parameter.

INSTRUMENTATION:

Type of instrumentation, used to perform the test. Automated continuous flow systems, consist of a sampler, peristaltic pump, manifold for reagent addition, detection system and readout system. Microcomputers are used to control the operation of analytical equipment and/or data acquisition.

REPORTING:

W and T are low level data qualifiers assigned to data that are near or below the detection limit values (3)(5). The code <W indicates that no measurable response was observed under the test conditions. The value reported indicates the minimum amount of analyte measured under routine conditions. W is usually less than the standard deviation of duplicates near zero. The code <T is used to represent a measurable amount of the analyte which under the test conditions is not verifiable. The reported result should be used only for large batches of similar data to evaluate background levels or trends of contaminants in the environment where more sensitive analytical methods are not available.

To provide a consistent Laboratory Services Branch approach to data reporting, the Water Quality Section calculates W from the standard deviation of duplicates ( $S_2$ ), near zero, by rounding down to the nearest 1,2 or 5 digit. T is five times W. The latest calculations, valid at date of publication for W and T values of all active methods, are contained in this report (APPENDIX B).

Data is reported to a maximum of three significant figures to the nearest W.

CALIBRATION:

The number of standards used to calibrate the analytical system plus blanks if applicable.

CONTROLS:

The calibration, drift, recovery, and interference controls that are used when applicable to ensure that the system is operating properly.

MODIFICATIONS:

Modifications to the test in 1994.

NOTES:

Explanatory notes which may aid the data user in interpreting results and information.

1.2 PERFORMANCE DATA SUMMARY PAGE

TITLE:

The name of the test parameter.

QUALITY CONTROL DATA FROM/TO:

The period of time over which data were collected.

LAB:

The laboratory in which the data was collected.

ANALYTICAL RANGE:

The full scale value for the analytical range is given in concentration units.

CALIBRATION CONTROL:

A table for the calibration control standards. The between run standard deviation ( $S$ ), the within run standard deviation ( $S_w$ ), the ratio  $S/S_w$ , and the ranges for acceptance limits of the control standards sums and differences.

RECOVERIES (Where applicable):

A table for the recovery control standards.

DUPLICATES:

A table of within run duplicate data. The data is sorted into a number of concentration spans. The coefficient of variation (%) is obtained by dividing the mean standard deviation ( $S_2$ ) for a particular concentration span by the mean concentration of duplicate results in that span and multiplying by 100.

OTHER CHECKS (Where applicable):

A table for other checks.

1.3 QUALITY CONTROL GRAPHICS PAGE

TITLE:

The name of the test parameter and unit of measurement.

DATE FROM/TO:

Period of time over which data were collected.

CALIBRATION CONTROL:

Calibration control standards sums and differences are plotted on a horizontal scale for the period of data collection (referred to on the graphs as "QUALITY CONTROL STANDARD A+B" for example). The vertical scale consists of the control limits expressed on either side of the expected value. Control limits were chosen from previous analytical performance data.

**PART 2.0**  
**CHEMISTRY**

## 2.1 Analytical Quality Control Program - Chemistry

Quality control is a continuous process that involves constant checks of sample processing. This report summarizes the QC data collected during analytical processing to monitor performance of the analytical system.

**Calibration** is conducted by analyzing a series of calibration standards covering the analytical range. Since a high degree of both precision and accuracy is required to detect and minimize any between-run changes, the standards are analyzed with as little handling as possible.

Once a system has been calibrated, quality control begins. Depending on the analytical procedure, quality control may be used to evaluate: calibration, blank, recovery, sensitivity, potential interference, and sample repeatability.

### Calibration and Blank

Calibration is controlled by a minimum of two quality control standards and a long term blank which are prepared and maintained independently of the calibration standards. The system is not calibrated with the quality control standards. The long term blank is prepared identical to the quality control standards but with zero concentration of the analyte. Control standards are prepared less frequently than calibration standards and errors in newly prepared calibration standards can be detected by this cross check. Newly prepared control standards are run in parallel with the old control standards and must meet control requirements over three consecutive runs before the new standards are accepted on line.

The standard deviation of the control standards is used to estimate the between run standard deviation ( $S$ ) and is compared against the within run standard deviation ( $S_w$ ). If the ratio  $S/S_w$  exceeds 1.5 then poor control of systematic error can be inferred (1). Values for  $S$  and  $S_w$  are calculated as follows:

$$2S^2 = (S_A)^2 + (S_B)^2$$

$$2S_w^2 = (S_{A-B})^2$$

Where

$S_A$  = standard deviation of control standard A

$S_B$  = standard deviation of control standard B

$S_{A-B}$  = standard deviation of the difference between control standards A and B

NOTE: If a second range is employed for a test, more control standards are used because, in many systems, the between run standard deviations are concentration dependent.

Detailed description of the quality control processes are outlined in several LSB reports (1)(2)(3).

### Control Limits

The control standards data are assessed and compared against the control limits established from previous data to determine whether the calibration process is in control. The control limits are examined yearly and may be adjusted if the method performance improves and/or the historical data base is increased. Control limits are calculated for the sums and differences of control standards (A,B,C,D) by the equations:

$$(A+B) \pm 4.0 \times S_{A+B} \text{ for the sum of A+B}$$

$$(B+C) \pm 4.0 \times S_{B+C} \text{ for the sum of B+C}$$

$$(C+D) \pm 4.0 \times S_{C+D} \text{ for the sum of C+D}$$

$$(A-B) \pm 3.0 \times S_{A-B} \text{ for the difference of A-B}$$

$$(B-C) \pm 3.0 \times S_{B-C} \text{ for the difference of B-C}$$

$$(C-D) \pm 3.0 \times S_{C-D} \text{ for the difference of C-D}$$

If a control limit is exceeded, the analysis is stopped, corrective action taken and the control standards are re-analyzed.

### Recovery

Some methods require sample pre-treatment, such as digestion or extraction. A recovery check, suitable to that method, is required to estimate the efficiency of the pre-treatment. Recovery standards are usually prepared at 0%, 20% and 80% of full scale. The solutions are analyzed in the same manner as routine samples. Although these solutions are not used to calibrate the instrument, corrections for the blank and matrix effects are calculated and applied if necessary. For an analytical run to be accepted, the recoveries should be within  $\pm(5\% + T/2)$  of their expected values. (T is defined in Appendix A). The average blank should be within three standard deviations of its historical mean. If a second range is employed for a test, at least one additional recovery standard is used.

### Sensitivity and Baseline

Any change in the sensitivity of the instrumentation is monitored periodically by analyzing a standard that is usually 80% of full scale, and comparing the peak height to the original calibration standards. Baseline drift is usually recorded by periodic analysis of pure deionized water (Pure-DW) which does not contain any of the analyte, but may be adjusted to correspond to sample pre-treatment.

### Interference

Interference checks are run on any test where a substance may be present in large enough concentration to affect the results. The checks are near the threshold concentration, beyond which the methodological safeguards used to minimize the interferences are no longer effective. These checks indicate that the interferences have no effect up to the specified concentrations. Spiked samples are not analyzed on a routine basis.

### Sample Repeatability

Generally, one sample out of twenty is run in duplicate up to a maximum of three per day. The samples are selected for non-adjacent, within-run duplicate analyses. By analyzing samples in duplicate, the ability of the analyst to obtain repeatable analytical results, within an analytical run, can be determined. For results to be acceptable, at least two-thirds of the duplicate data must conform to limits which are based on historical performance.

The observed differences in duplicate results are accumulated and sorted according to sample concentration span. A standard deviation is calculated for each sample concentration span. The algorithm differs from the conventional standard deviation as follows:

Conventional Std. Dev. (1)\*

Std. Dev. of Duplicates (2)\*

$$S_1 = \sqrt{\frac{\sum_{i=1}^n (\bar{x} - x_i)^2}{n-1}}$$

$$S_2 = \sqrt{\frac{\sum_{i=1}^{n'} (x_{1i} - x_{2i})^2}{2n'}}$$

\* Standard deviations used for the data summaries.

Where

$S_1$  = sample standard deviation

$S_2$  = duplicate difference standard deviation

$n$  = number of data

$\bar{x}$  = mean of data

$x_i$  =  $i^{\text{th}}$  result

$(x_1 - x_2)_i$  = difference of the  $i^{\text{th}}$  duplicate

$n'$  = number of duplicate pairs

Reported values for duplicate standard deviations have been treated by robust statistical methods (5)(6). The standard deviation ( $S_2$ ) of the duplicate difference is also expressed as the coefficient of variation (CV) using the untreated standard deviation.

$$CV = \frac{S_2}{\bar{X}} \times 100$$

## 2.2 PERFORMANCE SUMMARIES



## ACIDITY, GRAN

### IDENTIFICATION:

|                     |                                      |                   |                        |
|---------------------|--------------------------------------|-------------------|------------------------|
| Laboratory Unit     | Titration                            | Method Introduced | 01/08/82               |
| Method Reference No | E3248A                               | Units             | $\mu\text{g/L as H}^+$ |
| LIMS Product Code   | PHACD3248                            | Supervisor        | F. Lo                  |
| Sample Type/Matrix  | Precipitation, Throughfall, Stemflow |                   |                        |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 15 mL            |
| Container         | Glass or Plastic |

### ANALYTICAL PROCEDURE:

Sample aliquots (10.0 mL) are titrated with 0.01 N sodium hydroxide to pH >8.3. The titrant is standardized against 0.0005 N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. Data are subjected to Gran analysis.

pH and total fixed endpoint acidity are determined simultaneously.

### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

### REPORTING:

|                                |                    |                    |
|--------------------------------|--------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 1 | Current T value: 5 |
|--------------------------------|--------------------|--------------------|

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL (expected result is $16.6 \mu\text{g/L as H}^+$ ) plus 2 standards, e.g. QCA |
|-------------|---|

# ACIDITY, GRAN

QUALITY CONTROL DATA FROM 10/01/94 TO 14/12/94

Laboratory Unit: Titration

Analytical Range: to 1000 µg/L as H<sup>+</sup>

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 28 | 500.0                  | 501.4              | 1.4       | 4.1563                 |
| B:   | 28 | 200.0                  | 201.9              | 1.9       | 2.7800                 |
| A+B: | 28 | 700.0                  | 703.3              | 3.3       | 5.9977                 |
| A-B: | 28 | 300.0                  | 299.5              | -0.5      | 3.7464                 |

s.d.(AB) S(between runs): 3.54

Sw(within run): 2.65

S/Sw: 1.3

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

662 - 738 for A+B  
272 - 328 for A-B

## DUPLICATES:

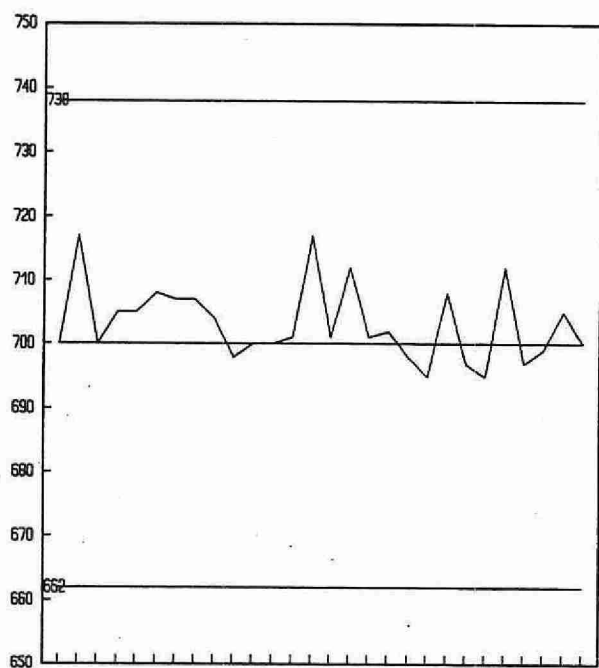
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 55           | 0 - 200                   | 2.7264                 | 4.3                         |
| 0            | 201 - 500                 | N.A.                   | N.A.                        |
| 0            | 501 - 1000                | N.A.                   | N.A.                        |
| 55           | Overall                   | 2.7264                 |                             |

## OTHER CHECKS:

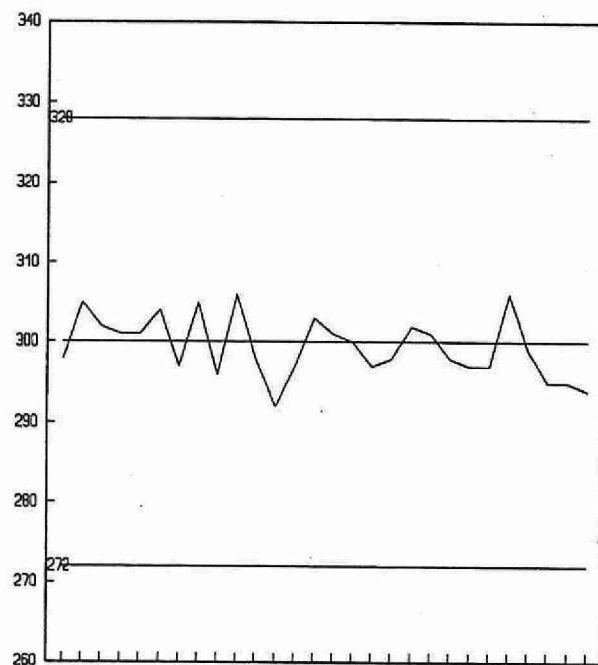
|                 | n  | Mean    | Standard Deviation (1) |
|-----------------|----|---------|------------------------|
| Long Term Blank | 27 | 14.7022 | 4.6020                 |

ACIDITY, GRAN ( $\mu\text{g/L as H}^+$ )

QUALITY CONTROL DATA FROM 10/01/94 TO 14/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## ACIDITY, TOTAL FIXED ENDPOINT

### IDENTIFICATION:

|                      |  |                   |                           |
|----------------------|--|-------------------|---------------------------|
| Laboratory Unit      | Titration  | Method Introduced | 01/08/82                  |
| Method Reference No. | E3248A   | Units             | mg/L as CaCO <sub>3</sub> |
| LIMS Product Code    | PHACD3248  | Supervisor        | F. Lo                     |
| Sample Type/Matrix   | Precipitation, Throughfall, Stemflow, Domestic Waters, Rivers, Lakes<br>(by special request: Industrial Waste, Sewage) |                   |                           |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 15 mL            |
| Container         | Glass or Plastic |

### ANALYTICAL PROCEDURE:

Sample aliquots (10.0 mL) are titrated with 0.01 N sodium hydroxide to pH >8.3. The titrant is standardized against 0.0005 N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. pH and gran acidity are determined simultaneously.

### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
|-------------|---------------------------------|

# ACIDITY, TOTAL FIXED ENDPOINT

QUALITY CONTROL DATA FROM 10/01/94 TO 14/12/94

Laboratory Unit: Titration

Analytical Range: to 100 mg/L as CaCO<sub>3</sub>

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 28 | 25.0                      | 25.12                 | 0.12      | 0.1946                    |
| B:   | 28 | 10.0                      | 10.13                 | 0.13      | 0.1374                    |
| A+B: | 28 | 35.0                      | 35.26                 | 0.26      | 0.2791                    |
| A-B: | 28 | 15.0                      | 14.99                 | -0.01     | 0.1887                    |

s.d.(AB) S(between runs): 0.17

Sw(within run): 0.13

S/Sw: 1.3

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

32.8 - 37.2 for A+B  
13.4 - 16.6 for A-B

## DUPLICATES:

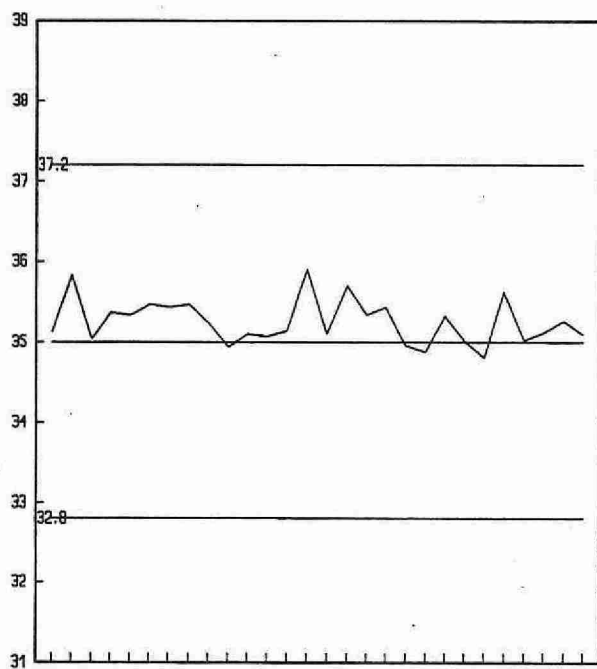
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 55              | 0.0 - 20.0                   | 0.1251                    | 4.1                            |
| 0               | 20.1 - 50.0                  | N.A.                      | N.A.                           |
| 0               | 50.1 - 100.0                 | N.A.                      | N.A.                           |
| 55              | Overall                      | 0.1251                    |                                |

## OTHER CHECKS:

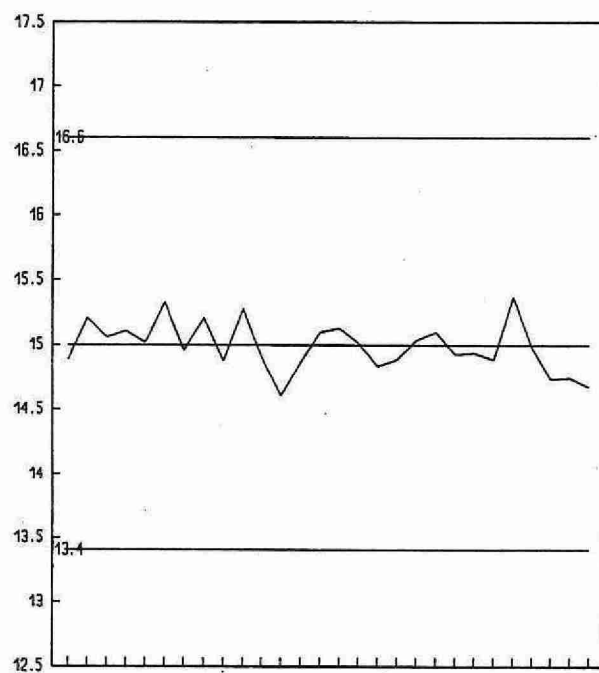
|                 | n  | Mean   | Standard<br>Deviation (1) |
|-----------------|----|--------|---------------------------|
| Long Term Blank | 27 | 0.7444 | 0.2159                    |

ACIDITY, TOTAL FIXED ENDPOINT (mg/L as CaCO<sub>3</sub>)

QUALITY CONTROL DATA FROM 10/01/94 TO 14/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## ALKALINITY, GRAN

### IDENTIFICATION:

|                      |   |                   |                           |
|----------------------|---|-------------------|---------------------------|
| Laboratory Unit      | Dorset                                      | Method Introduced | 26/07/79                  |
| LIS Test Name Code   | ALKTI                                       | Units             | mg/L as CaCO <sub>3</sub> |
| Work Station Code    | DOT   | Unit Code         | 064915                    |
| Method Code          | 0905T6                                      | Supervisor        | J. McBride                |
| Method Reference No. | E3042A                                      |                   |                           |
| Sample Type/Matrix:  | Streams, Lakes, Precipitation, Groundwaters |                   |                           |

### SAMPLING:

|                    |  |
|--------------------|--|
| Quantity Required: | 150 mL   |
| Container:         | 250 mL Amber polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred. |

### ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH <3.7. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subjected to Gran analysis.  
N.B. pH is determined simultaneously.

### INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data reduction software.

### REPORTING:

Maximum Significant Figures: 3

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

### CONTROLS:

|             |                                    |
|-------------|------------------------------------|
| Calibration | LTBL plus 4 standards, e.g. QCA    |
| Drift       | 2 standard buffers - 2 times daily |

# ALKALINITY, GRAN

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94

Laboratory Unit: Colourimetry

Full Scale: - to 100 mg/L as CaCO<sub>3</sub>

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 128 | 20.0                   | 19.998             | -0.002    | 0.2616                 |
| B:   | 128 | 5.0                    | 4.983              | -0.017    | 0.1482                 |
| C:   | 128 | -5.0                   | -4.976             | 0.024     | 0.3590                 |
| D:   | 128 | -1.25                  | -1.164             | 0.086     | 0.2279                 |
| A+B: | 128 | 25.0                   | 24.982             | -0.018    | 0.2426                 |
| A-B: | 128 | 15.0                   | 15.016             | 0.016     | 0.2247                 |
| C+D: | 128 | -6.25                  | -6.140             | 0.110     | 0.3775                 |
| C-D: | 128 | -3.75                  | -3.812             | -0.062    | 0.2761                 |

s.d.(AB) S(between runs): 0.21

Sw(within run): 0.16

S/Sw: 1.3

s.d.(CD) S(between runs): 0.30

Sw(within run): 0.20

S/Sw: 1.5

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 24.0  | - | 26.0  | for | A+B |
| 14.0  | - | 16.0  | for | A-B |
| -8.89 | - | -3.61 | for | C+D |
| -5.73 | - | -1.77 | for | C-D |

## DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 319          | -5.0 - 30.0               | 0.0979                 | 8.3                         |
| 5            | 30.1 - 60.0               | 2.3472                 | 8.6                         |
| 8            | 60.1 - 150.0              | 2.0261                 | 1.9                         |
| 43           | 151 - 300                 | 2.7541                 | 2.0                         |
| 375          | Overall                   | 0.1458                 |                             |

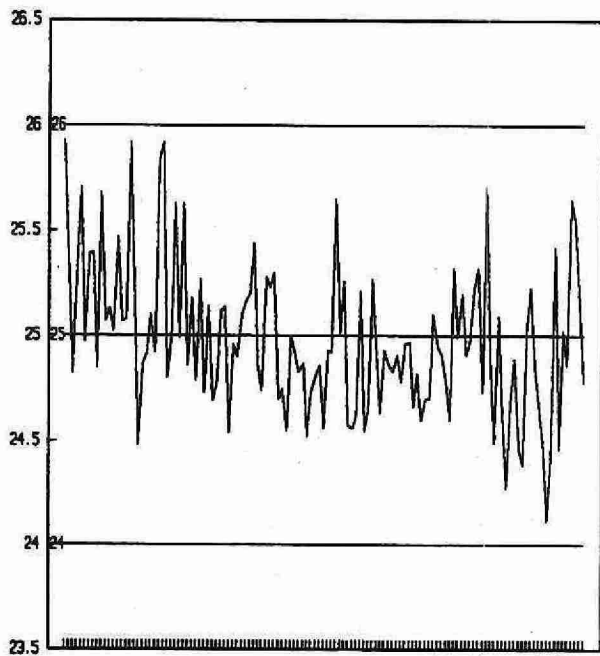
## OTHER CHECKS:

|                 | n   | Mean    | Standard Deviation (1) |
|-----------------|-----|---------|------------------------|
| Long Term Blank | 128 | -0.4305 | 0.1999                 |

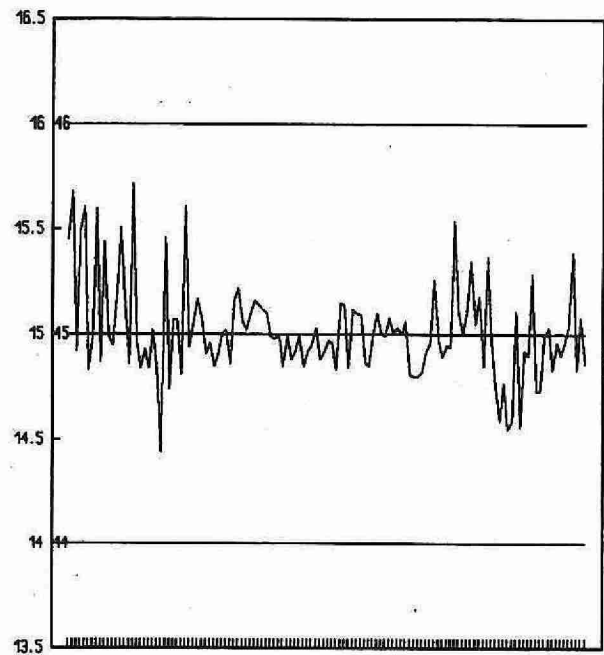


ALKALINITY, GRAN (mg/L as CaCO<sub>3</sub>)

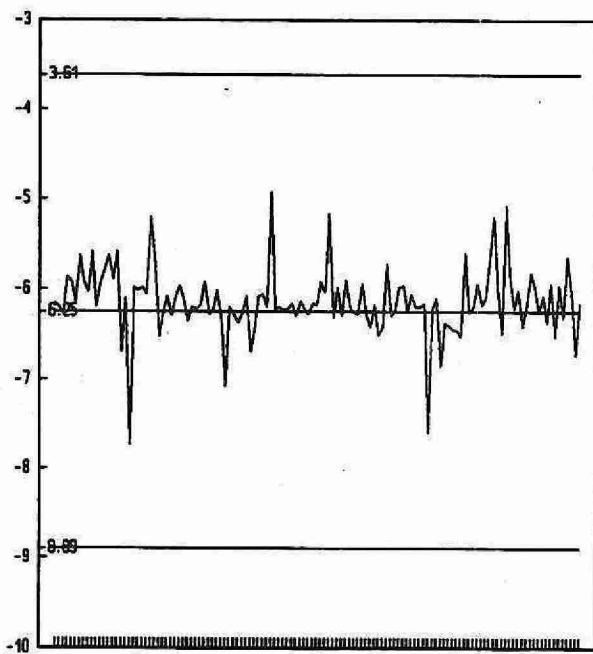
QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94



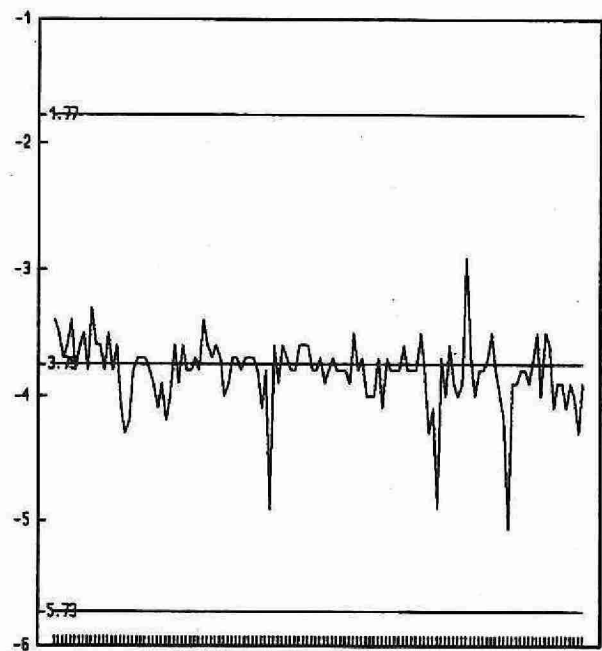
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

## ALKALINITY, GRAN

### IDENTIFICATION:

|                     |                              |                   |                           |
|---------------------|------------------------------|-------------------|---------------------------|
| Laboratory Unit     | Titration                    | Method Introduced | 09/07/80                  |
| Method Reference No | E3289A                       | Units             | mg/L as CaCO <sub>3</sub> |
| LIMS Product Code   | PHALG3289                    | Supervisor        | F. Lo                     |
| Sample Type/Matrix  | Rivers, Lakes, Precipitation |                   |                           |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or Plastic |

### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH <4.0. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subjected to Gran analysis.

pH, total fixed endpoint alkalinity, and conductivity are determined simultaneously.

### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

### REPORTING:

Maximum Significant Figures: 3

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | BL plus two standards, e.g. QCA                                 |
| Drift       | In run standards throughout the run (diluted tap water 20% V/V) |

# ALKALINITY, GRAN

QUALITY CONTROL DATA FROM 06/01/94 TO 03/10/94

Laboratory Unit: Titration

Analytical Range: to 25.0 mg/L as CaCO<sub>3</sub>

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| C:   | 14 | 10.0                      | 10.034                | 0.034     | 0.1587                    |
| D:   | 14 | 2.5                       | 2.421                 | -0.079    | 0.1164                    |
| C+D: | 14 | 12.5                      | 12.454                | -0.046    | 0.1017                    |
| C-D: | 14 | 7.5                       | 7.613                 | 0.113     | 0.2591                    |

s.d.(AB)

S(between runs): 0.14

Sw(within run): 0.18

S/Sw: 0.8

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

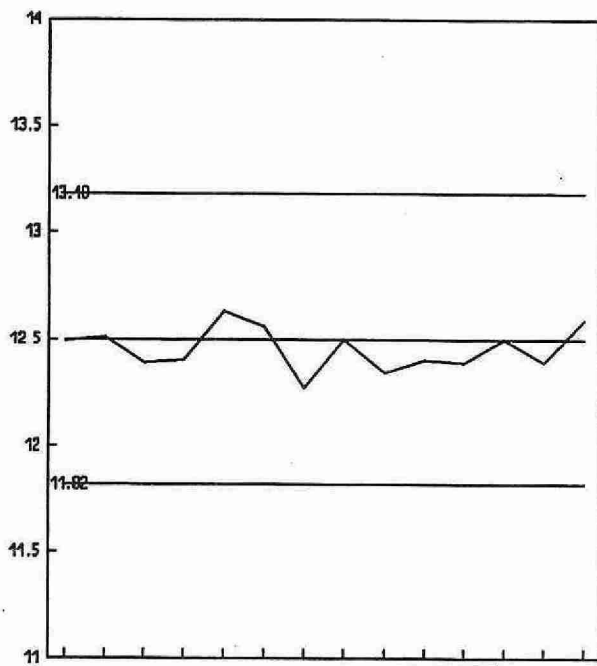
11.82 - 13.18 for C+D  
6.99 - 8.01 for C-D

## DUPLICATES:

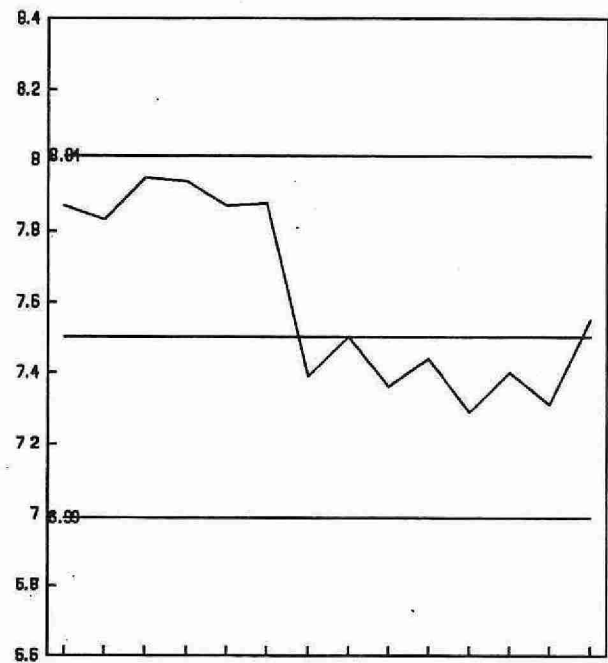
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 5               | -2.0 - 10.0                  | 0.1213                    | 23.2                           |
| 2               | 10.1 - 25.0                  | N.A.                      | N.A.                           |
| 7               | Overall                      | 0.1093                    |                                |

ALKALINITY, GRAN (mg/L as CaCO<sub>3</sub>)

QUALITY CONTROL DATA FROM 06/01/94 TO 03/10/94



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

## ALKALINITY, TOTAL FIXED ENDPOINT

### IDENTIFICATION:

|                      |   |                   |                           |
|----------------------|---|-------------------|---------------------------|
| Laboratory Unit      | Dorset                                      | Method Introduced | 26/07/79                  |
| LIS Test Name Code   | ALKT  | Units             | mg/L as CaCO <sub>3</sub> |
| Work Station Code    | DOT   | Unit Code         | 064915                    |
| Method Code          | 0905T3                                      | Supervisor        | J. McBride                |
| Method Reference No. | E3042A                                      |                   |                           |
| Sample Type/Matrix:  | Streams, Lakes, Precipitation, Groundwaters |                   |                           |

### SAMPLING:

|                    |  |
|--------------------|--|
| Quantity Required: | 150 mL   |
| Container:         | 250 mL Amber polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred. |

### ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

### INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data processing software.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift       | 2 standard buffers - once daily |

# ALKALINITY, TOTAL FIXED ENDPOINT

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94

Laboratory : Dorset

Analytical Range: 100 mg/L as CaCO<sub>3</sub>

## CALIBRATION CONTROL:

|      | n   | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|-----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 128 | 20.0                      | 19.8                  | -0.2      | 0.2290                    |
| B:   | 128 | 5.00                      | 4.9                   | -0.1      | 0.1229                    |
| A+B: | 128 | 25.0                      | 24.6                  | -0.4      | 0.3163                    |
| A-B: | 128 | 15.0                      | 14.8                  | -0.2      | 0.1879                    |

s.d.(AB) S(between runs): 0.18

Sw:(within run): 0.13

S/Sw: 1.4

On any given day the calibration is accepted if the values obtained lie within the ranges:

24.0 - 26.0 for A+B  
14.0 - 16.0 for A-B

## DUPLICATES:

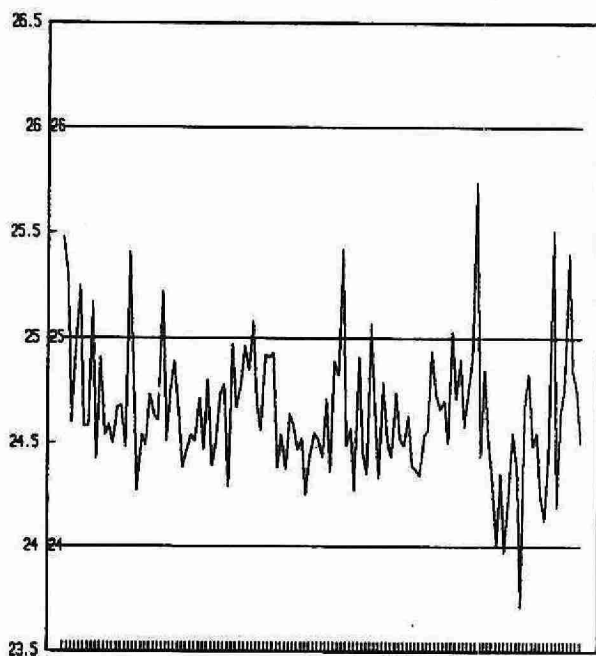
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 284             | 0.0 - 30.0                   | 0.0700                    | 3.5                            |
| 5               | 30.1 - 60.0                  | 0.8287                    | 8.0                            |
| 8               | 60.1 - 150                   | 0.3522                    | 0.3                            |
| 43              | 151 - 300                    | 0.7719                    | 1.3                            |
| 340             | Overall                      | 0.1053                    |                                |

## OTHER CHECKS:

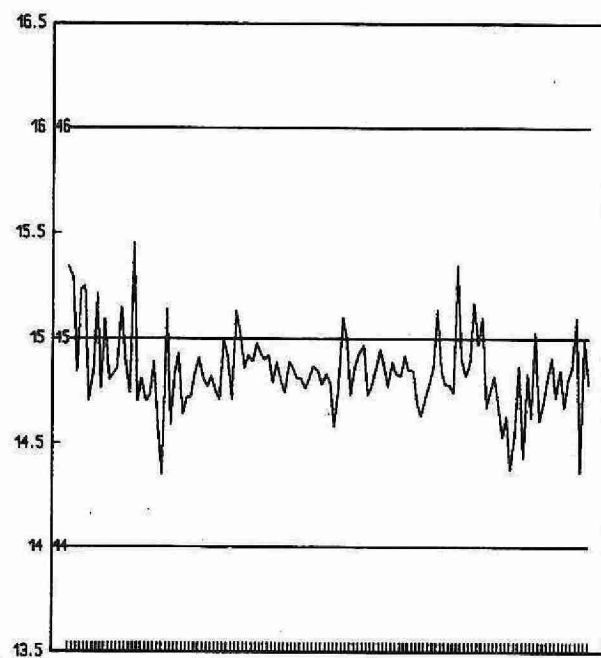
|                 | n   | Mean  | Standard<br>Deviation (1) |
|-----------------|-----|-------|---------------------------|
| Long Term Blank | 128 | 1.332 | 0.3301                    |

ALKALINITY, TOTAL FIXED ENDPOINT (mg/L as  $\text{CaCO}_3$ )

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## ALKALINITY, TOTAL FIXED ENDPOINT

### IDENTIFICATION:

|                      |                                    |                   |                           |
|----------------------|------------------------------------|-------------------|---------------------------|
| Laboratory Unit      | Titration                          | Method Introduced | 09/07/80                  |
| Method Reference No. | E3218A                             | Units             | mg/L as CaCO <sub>3</sub> |
| LIMS Product Code    | PHALK3218                          | Supervisor        | F. Lo                     |
| Sample Type/Matrix   | Domestic Waters, Sewage, Effluents |                   |                           |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or Plastic |

### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH endpoint of 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

pH, and conductivity are determined simultaneously.

### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | BL plus 4 standards, e.g. QCA                                      |
| Drift       | In run standards throughout the run (tap water diluted to 50% V/V) |



# ALKALINITY, TOTAL FIXED ENDPOINT

QUALITY CONTROL DATA FROM 04/01/94 TO 21/12/94

Laboratory Unit: Titration

Analytical Range: to 1000 mg/L as CaCO<sub>3</sub>

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 80 | 250                       | 250.04                | 0.04      | 1.3885                    |
| B:   | 80 | 100                       | 100.48                | 0.48      | 0.4481                    |
| C:   | 80 | 100                       | 99.57                 | -0.43     | 0.7953                    |
| D:   | 80 | 25                        | 24.73                 | -0.27     | 0.2943                    |
| A+B: | 80 | 350                       | 350.52                | 0.52      | 1.6591                    |
| A-B: | 80 | 150                       | 149.56                | -0.44     | 1.2245                    |
| C+D: | 80 | 125                       | 124.30                | -0.70     | 1.0051                    |
| C-D: | 80 | 75                        | 74.84                 | -0.16     | 0.6542                    |

s.d.(AB)    S(between runs): 1.03                      Sw(within run): 0.87                      S/Sw: 1.2

s.d.(CD)    S(between runs): 0.60                      Sw(within run): 0.46                      S/Sw: 1.3

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

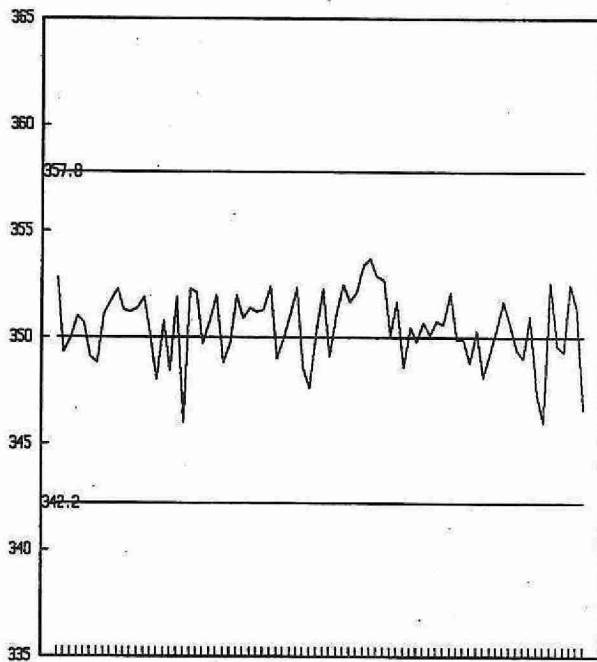
|        |   |        |     |     |
|--------|---|--------|-----|-----|
| 342.20 | - | 357.80 | for | A+B |
| 144.15 | - | 155.85 | for | A-B |
| 119.84 | - | 130.16 | for | C+D |
| 71.13  | - | 78.87  | for | C-D |

## DUPLICATES:

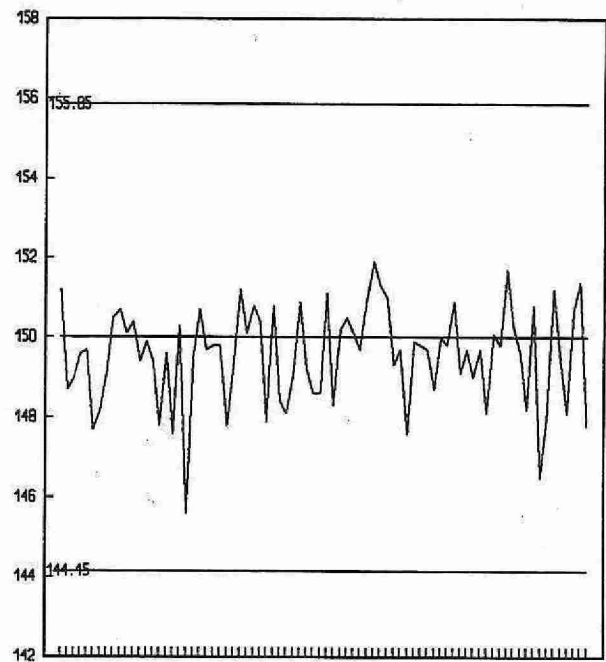
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 79              | 0 - 100                      | 0.5713                    | 0.8                            |
| 65              | 101 - 200                    | 0.8676                    | 0.6                            |
| 61              | 201 - 500                    | 1.4463                    | 0.6                            |
| 0               | 501 - 1000                   | N.A.                      | N.A.                           |
| 205             | Overall                      | 0.8953                    |                                |

**ALKALINITY, TOTAL FIXED ENDPOINT** (mg/L as CaCO<sub>3</sub>)

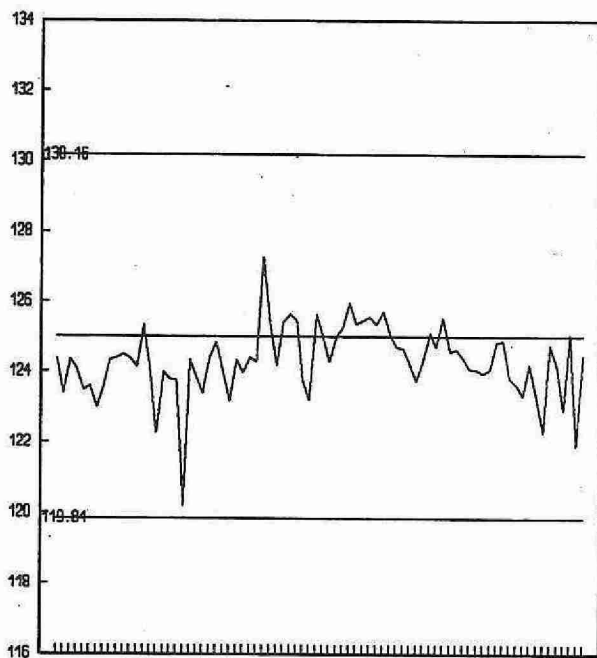
QUALITY CONTROL DATA FROM 04/01/94 TO 21/12/94



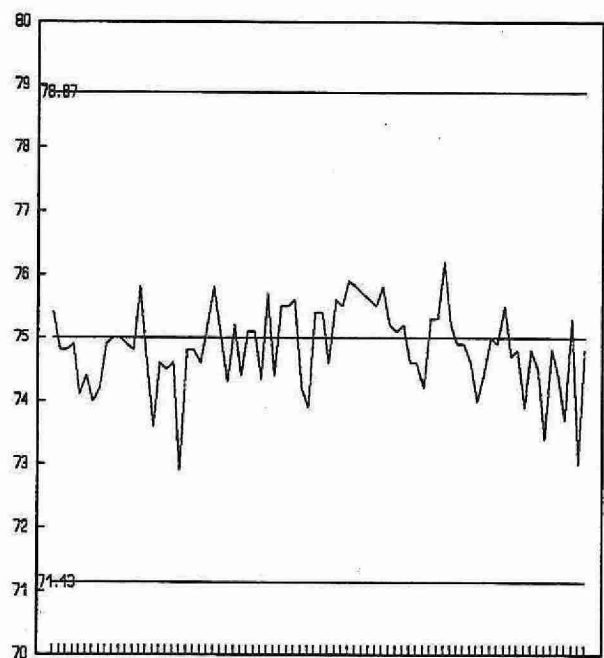
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

## ALKALINITY, TOTAL FIXED ENDPOINT

### IDENTIFICATION:

|                      |                    |                   |                           |
|----------------------|--------------------|-------------------|---------------------------|
| Laboratory Unit      | Titration          | Method Introduced | Before 1980               |
| Method Reference No. | E3228A             | Units             | mg/L as CaCO <sub>3</sub> |
| LIMS Product Code    | PHALK3228          | Supervisor        | F. Lo                     |
| Sample Type/Matrix   | Landfill leachates |                   |                           |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or Plastic |

### ANALYTICAL PROCEDURE:

Samples are pipetted manually (50.0 mL) and titrated with 0.02 N sulphuric acid to pH endpoint of 4.5. Analysis is performed on the supernatant or filtrate.

### INSTRUMENTATION:

Automated modular titration system.

### REPORTING:

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |
|--------------------------------|----------------------|----------------------|

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | BL plus 2 standards, e.g. QCA                        |
| Drift       | In run standards throughout the run (100% tap water) |

# ALKALINITY, TOTAL FIXED ENDPOINT

QUALITY CONTROL DATA FROM 22/02/94 TO 14/12/94

Laboratory Unit: Titration

Analytical Range: to 1000 mg/L as  $\text{CaCO}_3$

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 21 | 570.0                  | 570.7              | 0.7       | 1.9312                 |
| B:   | 21 | 114.0                  | 116.5              | 2.5       | 0.6388                 |
| A+B: | 21 | 684.0                  | 687.2              | 3.2       | 2.0445                 |
| A-B: | 21 | 456.0                  | 454.3              | -1.7      | 2.0235                 |

s.d.(AB) S(between runs): 1.44

Sw(within run): 1.43

S/Sw: 1.01

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

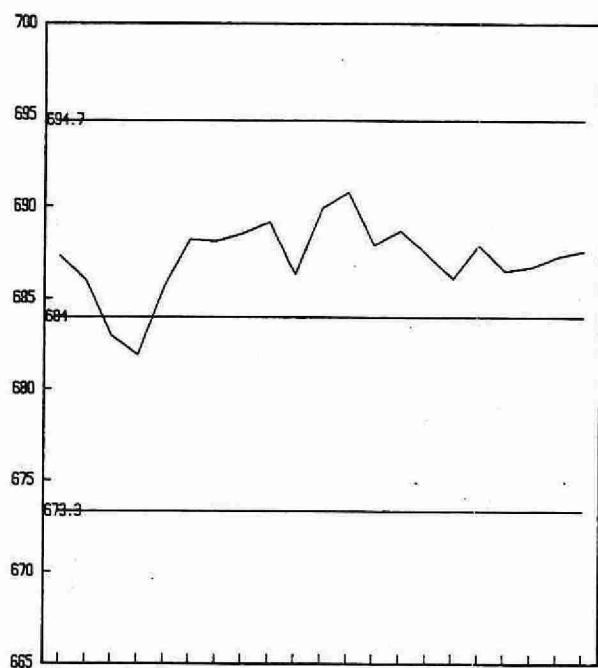
673.3 - 694.7 for A+B  
448 - 464 for A-B

## DUPLICATES:

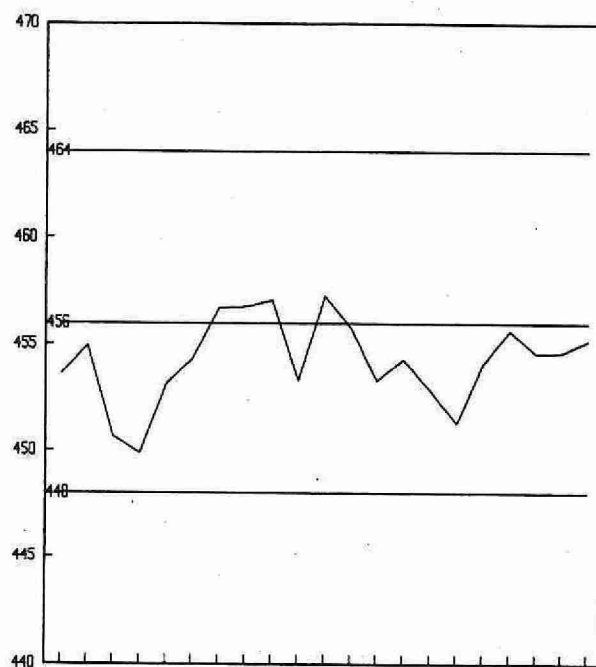
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 25           | 0 - 200                   | 0.3527                 | 0.6                         |
| 34           | 201 - 500                 | 1.1216                 | 0.8                         |
| 4            | 501 - 1000                | 4.8885                 | 0.6                         |
| 63           | Overall                   | 0.9786                 |                             |

ALKALINITY, TOTAL FIXED ENDPOINT (mg/L as CaCO<sub>3</sub>)

QUALITY CONTROL DATA FROM 22/02/94 TO 14/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## ALKALINITY, TOTAL FIXED ENDPOINT

### IDENTIFICATION:

|                      |                              |                   |                           |
|----------------------|------------------------------|-------------------|---------------------------|
| Laboratory Unit      | Titration                    | Method Introduced | 09/07/80                  |
| Method Reference No. | E3289A                       | Units             | mg/L as CaCO <sub>3</sub> |
| LIMS Product Code    | PHALK3289                    | Supervisor        | F. Lo                     |
| Sample Type/Matrix   | Rivers, Lakes, Precipitation |                   |                           |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or Plastic |

### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH <4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

pH, Gran alkalinity, and conductivity are determined simultaneously.

### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | BL plus 4 standards, e.g. QCA                                      |
| Drift       | In run standards throughout the run (tap water diluted to 20% V/V) |

# ALKALINITY, TOTAL FIXED ENDPOINT

QUALITY CONTROL DATA FROM 06/01/94 TO 12/12/94

Laboratory Unit: Titration

Analytical Range: to 1000 mg/L as CaCO<sub>3</sub>

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 82 | 250.0                     | 250.84                | 0.84      | 1.335                     |
| B:   | 82 | 50.0                      | 50.24                 | 0.24      | 0.433                     |
| C:   | 82 | 10.0                      | 9.98                  | -0.02     | 0.172                     |
| D:   | 82 | 2.5                       | 2.52                  | 0.02      | 0.080                     |
| A+B: | 82 | 300.0                     | 301.08                | 1.08      | 1.602                     |
| A-B: | 82 | 200.0                     | 200.60                | 0.60      | 1.171                     |
| C+D: | 82 | 12.5                      | 12.51                 | 0.01      | 0.175                     |
| C-D: | 82 | 7.5                       | 7.46                  | -0.04     | 0.203                     |

s.d.(AB) S(between runs): 0.99 Sw(within run): 0.83 S/Sw: 1.2

s.d.(BC) S(between runs): 0.13 Sw(within run): 0.14 S/Sw: 0.9

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

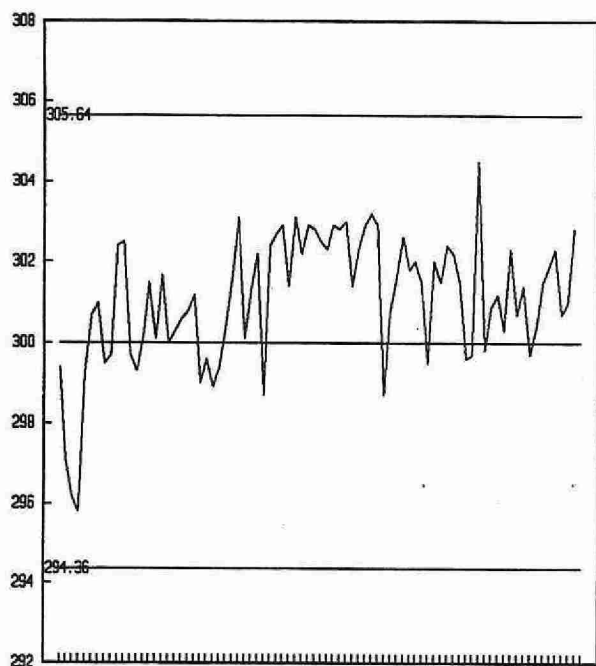
|        |   |        |     |     |
|--------|---|--------|-----|-----|
| 294.36 | - | 305.64 | for | A+B |
| 195.77 | - | 204.23 | for | A-B |
| 11.76  | - | 13.24  | for | C+D |
| 6.95   | - | 8.05   | for | C-D |

## DUPLICATES:

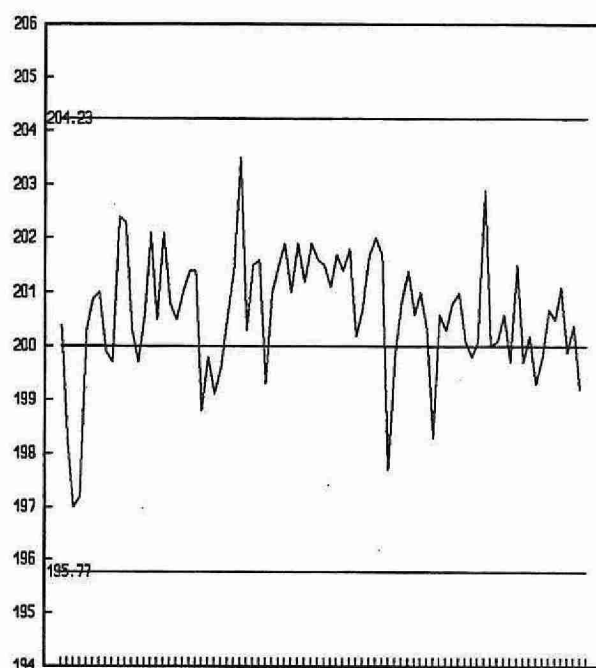
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 36              | 0 - 50                       | 0.1729                    | 1.6                            |
| 38              | 50 - 100                     | 0.4023                    | 0.6                            |
| 149             | 101 - 300                    | 0.6428                    | 0.4                            |
| 2               | 301 - 1000                   | N.A.                      | N.A.                           |
| 225             | Overall                      | 0.5289                    |                                |

**ALKALINITY, TOTAL FIXED ENDPOINT** (mg/L as CaCO<sub>3</sub>)

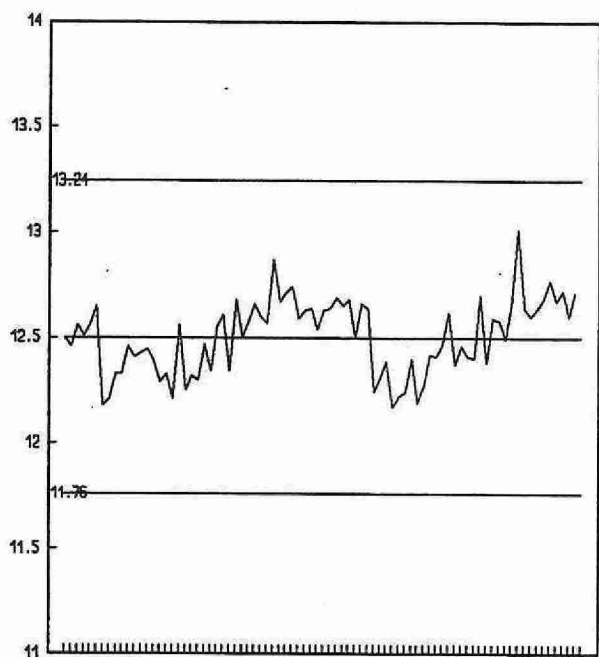
QUALITY CONTROL DATA FROM 06/01/94 TO 12/12/94



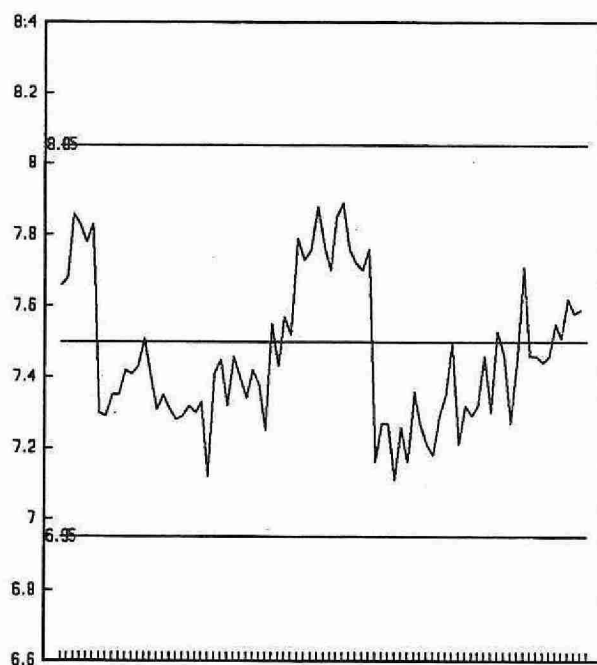
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT



## ALKALINITY, TOTAL FIXED ENDPOINT

### IDENTIFICATION:

|                      |   |                   |                           |
|----------------------|---|-------------------|---------------------------|
| Laboratory Unit      | Dorset                                      | Method Introduced | 21/10/85                  |
| LIS Test Name Code   | ALKT3                                       | Units             | mg/L as CaCO <sub>3</sub> |
| Work Station Code    | DOT   | Unit Code         | 064915                    |
| Method Code          | 0905T3                                      | Supervisor        | J. McBride                |
| Method Reference No. | E3042A                                      |                   |                           |
| Sample Type/Matrix   | Streams, Lakes, Precipitation, Groundwaters |                   |                           |

### SAMPLING:

|                   |   |
|-------------------|---|
| Quantity Required | 150 mL  |
| Container         | Amber polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred. |

### ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH 3.8. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

### INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data processing software.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift       | 2 standard buffers - once daily |

# ALKALINITY, TOTAL FIXED ENDPOINT

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94

Laboratory : Dorset

Analytical Range: 100 mg/L as CaCO<sub>3</sub>

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 128 | 20.0                   | 20.1               | 0.1       | 0.3707                 |
| B:   | 128 | 5.00                   | 4.995              | -0.005    | 0.2679                 |
| A+B: | 128 | 25.0                   | 25.1               | 0.1       | 0.5775                 |
| A-B: | 128 | 15.0                   | 15.1               | 0.1       | 0.2912                 |

s.d.(AB) S(between runs): 0.32

Sw:(within run): 0.21

S/Sw: 1.6

On any given day the calibration is accepted if the values obtained lie within the ranges:

23.28 - 26.72 for A+B  
13.5 - 16.5 for A-B

## DUPLICATES:

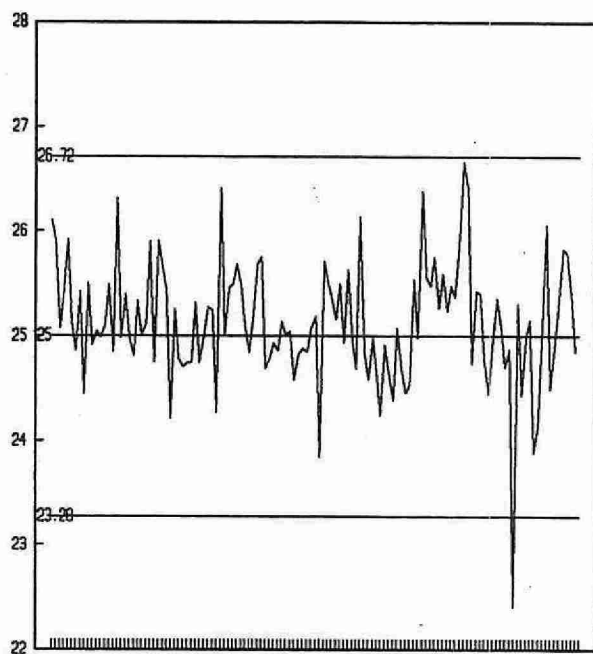
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 318          | 0.0 - 30.0                | 0.2749                 | 4.3                         |
| 5            | 30.1 - 60.0               | 1.4654                 | 17.4                        |
| 6            | 60.1 - 150                | 0.7024                 | 0.6                         |
| 46           | 151 - 300                 | 0.9255                 | 1.2                         |
| 375          | Overall                   | 0.3514                 |                             |

## OTHER CHECKS:

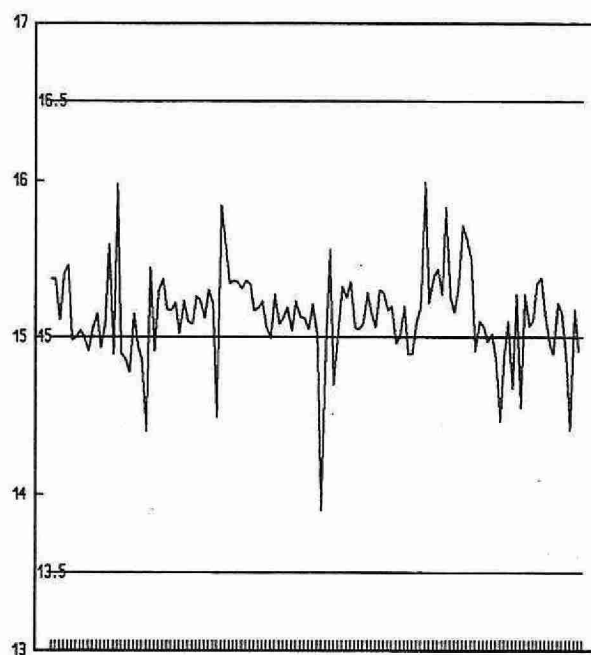
|                 | n   | Mean  | Standard Deviation (1) |
|-----------------|-----|-------|------------------------|
| Long Term Blank | 128 | 7.508 | 1.2528                 |

ALKALINITY, TOTAL FIXED ENDPOINT (mg/L as  $\text{CaCO}_3$ )

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## ALUMINUM, REACTIVE SPECIES

### IDENTIFICATION:

|                      |                                    |                   |            |
|----------------------|------------------------------------|-------------------|------------|
| Laboratory Unit      | Dorset                             | Method Introduced | 24/10/85   |
| LIS Test Name Code   | ALNDCV                             | Units             | µg/L as Al |
| Work Station Code    | DOALSP                             | Unit Code         | 063813     |
| Method Code          | 0928C2                             | Supervisor        | J. McBride |
| Method Reference No. | E3020A                             |                   |            |
| Sample Type/Matrix   | Streams, Lakes, and Soil Leachates |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 30 mL            |
| Container         | Plastic or glass |

### ANALYTICAL PROCEDURE:

The procedure is based on the formation of an aluminum catechol-violet complex at pH 6.2. Phenanthroline hydroxylamine HCl reagents are used to reduce interference by iron. An ion exchange column is used for separating organic and inorganic aluminum. Concentrations of aluminum are determined by comparison with a similarly prepared series of standards and reported as µg/L as CV reactive Al.

### INSTRUMENTATION:

Automated auto-analyzer/sampler system with colourimeter and chart recorder.

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |
|--------------------------------|--------------------|---------------------|

### CALIBRATION:

BL plus 10 standards daily

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 standards, e.g. QCA                                 |
| Drift       | BL every 10 samples and BL plus check standard every 20 samples |

### NOTES:

Jan. 1995 LIMS replaced LIS and the method reference no. was changed from E3020A to E3375A. LIMS product code is ALSP3375.

**ALUMINUM, REACTIVE SPECIES  
(ALNDCV)**

QUALITY CONTROL DATA FROM 18/01/94 TO 23/12/94

Laboratory: Dorset

Full Scale: to 1000 µg/L as Al

**CALIBRATION CONTROL:**

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 41 | 600                       | 593.85                | -6.15     | 5.9058                    |
| B:   | 41 | 200                       | 198.12                | -1.88     | 2.8913                    |
| C:   | 41 | 60                        | 58.63                 | -1.37     | 2.2108                    |
| A+B: | 41 | 800                       | 791.98                | -8.02     | 8.1286                    |
| A-B: | 41 | 400                       | 395.73                | -4.27     | 4.5168                    |
| B+C: | 41 | 260                       | 256.76                | -3.24     | 3.7402                    |
| B-C: | 41 | 140                       | 139.49                | -0.51     | 3.5364                    |

s.d.(AB)      S(between runs): 4.65

Sw(within run): 3.19

S/Sw: 1.5

s.d.(BC)      S(between runs): 2.57

Sw(within run): 2.50

S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

|     |   |     |         |
|-----|---|-----|---------|
| 775 | - | 825 | for A+B |
| 390 | - | 410 | for A-B |
| 252 | - | 268 | for B+C |
| 134 | - | 146 | for B-C |

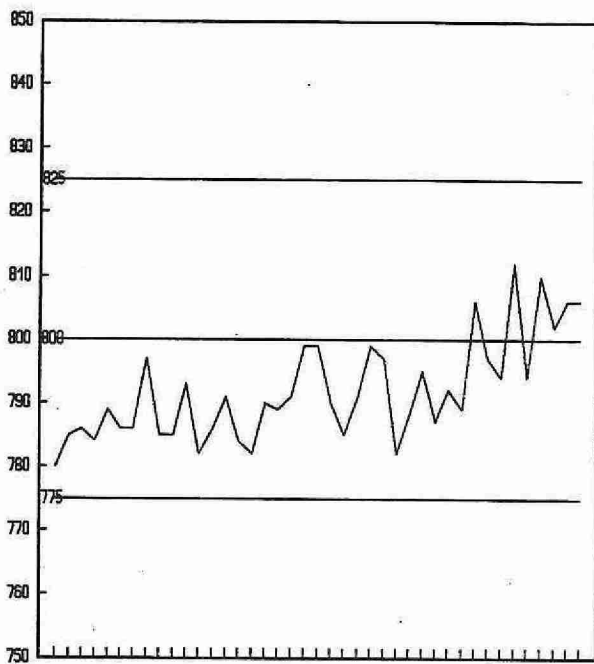
**DUPLICATES:**

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 72              | 0.0 - 100                    | 2.9633                    | 6.1                            |
| 30              | 101 - 200                    | 3.6962                    | 3.1                            |
| 15              | 201 - 500                    | 12.8750                   | 4.0                            |
| 2               | 501 - 1000                   | N.A.                      | N.A.                           |
| 119             | Overall                      | 3.7210                    |                                |

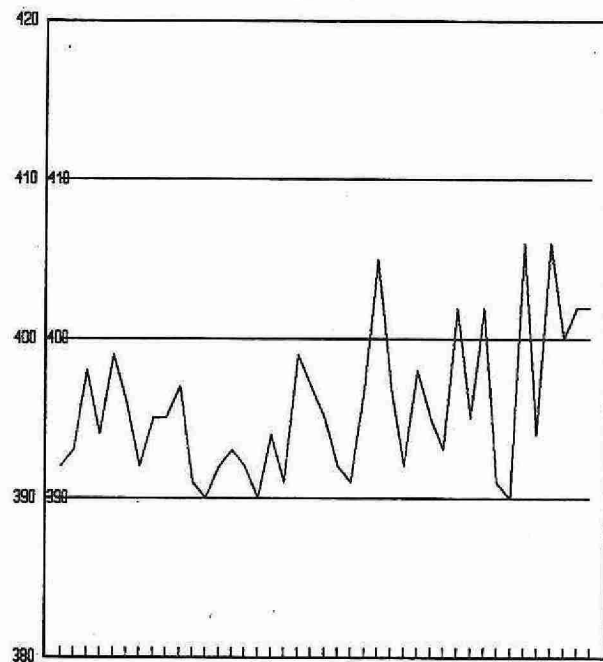
**OTHER CHECKS:**

|                 | n  | Mean  | Standard<br>Deviation (1) |
|-----------------|----|-------|---------------------------|
| Long Term Blank | 41 | 0.293 | 1.0306                    |

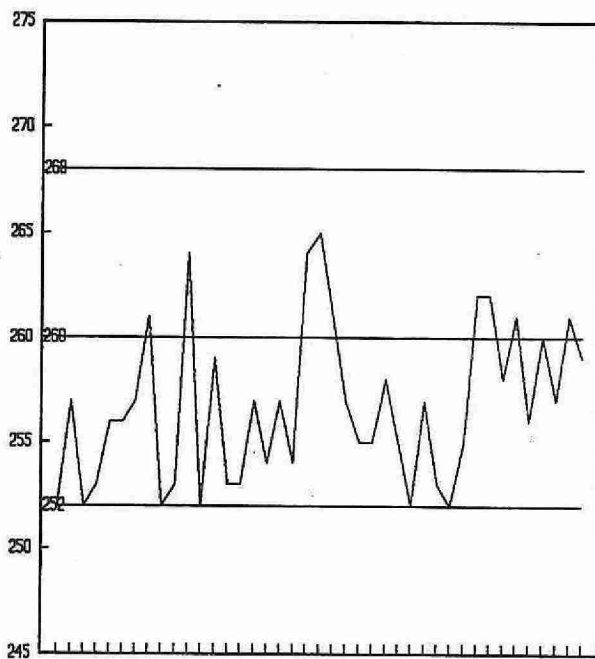
**ALUMINUM, REACTIVE SPECIES** (µg/L as Al)  
(ALNDCV)  
QUALITY CONTROL DATA FROM 18/01/94 TO 23/12/94



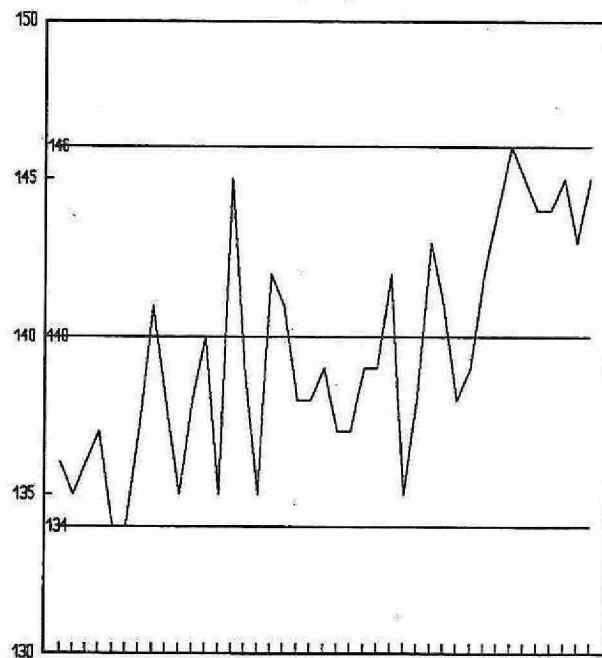
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## ALUMINUM, REACTIVE SPECIES

### IDENTIFICATION:

|                      |                                    |                   |            |
|----------------------|------------------------------------|-------------------|------------|
| Laboratory Unit      | Dorset                             | Method Introduced | 24/10/85   |
| LIS Test Name Code   | ALEXCV                             | Units             | µg/L as Al |
| Work Station Code    | DOALSP                             | Unit Code         | 063813     |
| Method Code          | 0928C2                             | Supervisor        | J. McBride |
| Method Reference No. | E3256A                             |                   |            |
| Sample Type/Matrix   | Streams, Lakes, and Soil Leachates |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 30 mL            |
| Container         | Plastic or glass |

### ANALYTICAL PROCEDURE:

The procedure is based on the formation of an aluminum catechol-violet complex at pH 6.2. Phenanthroline hydroxylamine HCl reagents are used to reduce interference by iron. An ion exchange column is used for separating organic and inorganic aluminum. Concentrations of aluminum are determined by comparison with a similarly prepared series of standards and reported as µg/L as CV reactive Al.

### INSTRUMENTATION:

Automated auto-analyzer/sampler system with colourimeter and chart recorder.

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |
|--------------------------------|--------------------|---------------------|

### CALIBRATION:

BL plus 10 standards daily

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 standards, e.g. QCA                                 |
| Drift       | BL every 10 samples and BL plus check standard every 20 samples |

### NOTES:

Jan. 1995 LIMS replaced LIS and the method reference no. was changed from E3256A to E3375A. LIMS product code is ALSP3375.

**ALUMINUM, REACTIVE SPECIES  
(ALEXCV)**

QUALITY CONTROL DATA FROM 18/01/94 TO 23/12/94

Laboratory: Dorset

Full Scale: to 1000 µg/L as Al

**CALIBRATION CONTROL:**

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 40 | 600                       | 594.13                | -5.87     | 5.9147                    |
| B:   | 40 | 200                       | 197.83                | -2.17     | 3.1288                    |
| C:   | 40 | 60                        | 59.30                 | -0.70     | 1.9768                    |
| A+B: | 40 | 800                       | 791.95                | -8.05     | 8.1805                    |
| A-B: | 40 | 400                       | 396.30                | -3.70     | 4.7566                    |
| B+C: | 40 | 260                       | 257.13                | -2.87     | 4.0204                    |
| B-C: | 40 | 140                       | 138.53                | -1.47     | 3.3511                    |

s.d.(AB) S(between runs): 4.73

Sw(within run): 3.36

S/Sw: 1.4

s.d.(BC) S(between runs): 2.62

Sw(within run): 2.37

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|     |   |     |     |     |
|-----|---|-----|-----|-----|
| 775 | - | 825 | for | A+B |
| 385 | - | 415 | for | A-B |
| 252 | - | 268 | for | B+C |
| 134 | - | 146 | for | B-C |

**DUPLICATES:**

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 90              | 0.0 - 100                    | 3.9829                    | 9.5                            |
| 22              | 101 - 200                    | 6.7171                    | 6.4                            |
| 3               | 201 - 500                    | 15.9463                   | 5.5                            |
| 1               | 501 - 1000                   | N.A.                      | N.A.                           |
| 116             | Overall                      | 4.6497                    |                                |

**OTHER CHECKS:**

|                 | n  | Mean  | Standard<br>Deviation (1) |
|-----------------|----|-------|---------------------------|
| Long Term Blank | 40 | 0.325 | 0.8286                    |

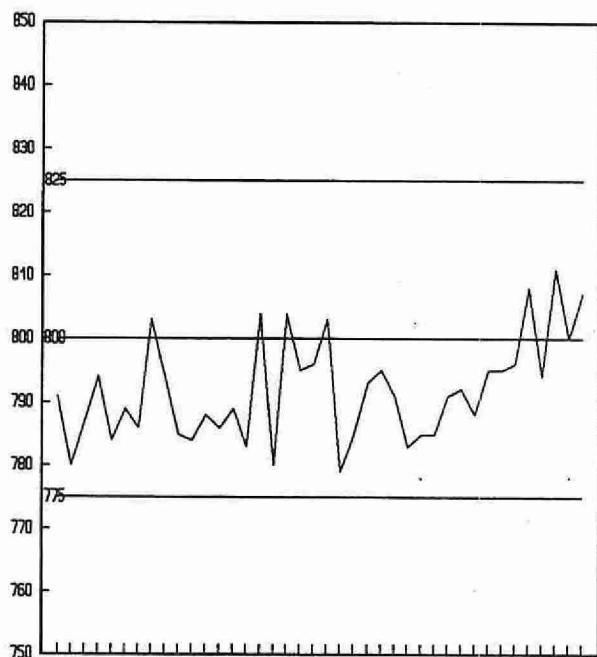


# ALUMINUM, REACTIVE SPECIES

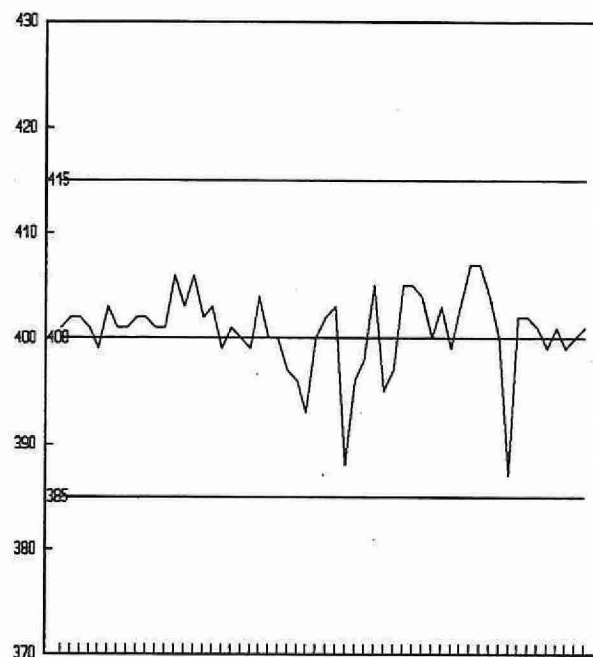
( $\mu\text{g/L}$  as AL)

(ALEXCV)

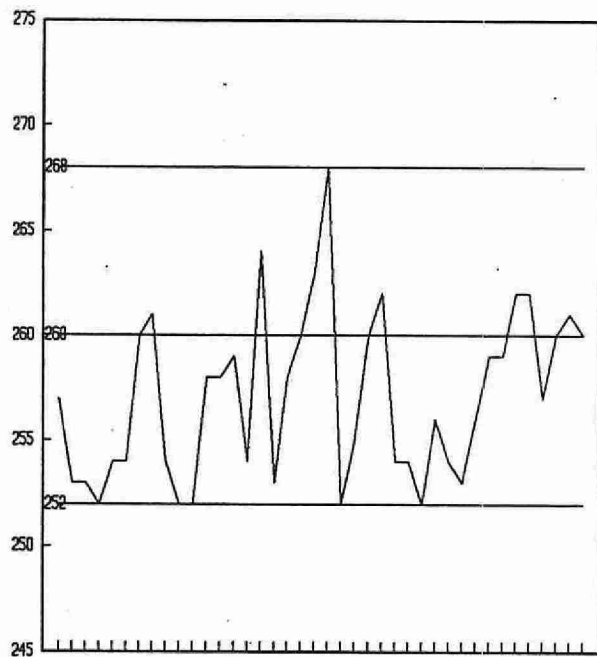
QUALITY CONTROL DATA FROM 18/01/94 TO 23/12/94



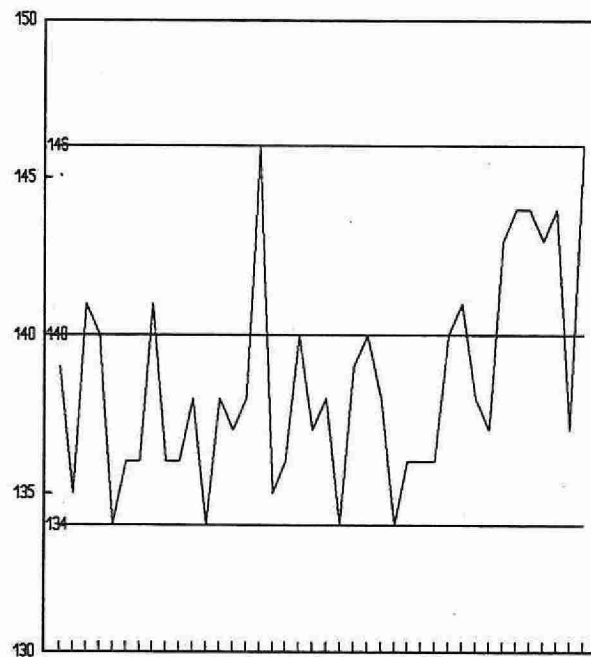
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## ALUMINUM, TOTAL

### IDENTIFICATION:

|                      |   |                   |            |
|----------------------|---|-------------------|------------|
| Laboratory Unit      | Dorset  | Method Introduced | 06/09/83   |
| LIS Test Name Code   | ALUT  | Units             | µg/L as Al |
| Work Station Code    | DOAL  | Unit Code         | 063813     |
| Method Code          | 005AF2  | Supervisor        | J. McBride |
| Method Reference No. | E3300A  |                   |            |
| Sample Type/Matrix   | Streams, Lakes, Precipitation, Biota and Groundwaters |                   |            |

### SAMPLING:

|                   |   |
|-------------------|---|
| Quantity Required | 10 mL   |
| Container         | 100 mL Polypropylene bottle capped, acidified to 0.1% with HNO <sub>3</sub> |

### ANALYTICAL PROCEDURE:

This procedure is based on the formation of an aluminum-catechol violet complex at pH 6.2. Acidified samples are oxidized by UV digestion for total aluminum. Phenanthroline-hydroxylamine-HCL reagents are used to reduce interference by iron. Concentrations of aluminum are determined by comparison with a similarly prepared series of standards.

### INSTRUMENTATION:

UV-digestor

An autoanalyzer with microprocessor for DCI system.

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |
|--------------------------------|--------------------|---------------------|

### CALIBRATION:

BL plus 8 standards daily

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 standards, e.g. QCA                                 |
| Drift       | BL every 10 samples and BL plus check standard every 20 samples |

# ALUMINUM, TOTAL

QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94

Laboratory: Dorset

Full Scale: to 1000 µg/L as Al

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 56 | 600                    | 603.39             | 3.39      | 4.4382                 |
| B:   | 56 | 200                    | 202.71             | 2.71      | 1.6036                 |
| C:   | 56 | 60                     | 60.21              | 0.21      | 2.0689                 |
| A+B: | 56 | 800                    | 806.11             | 6.11      | 5.4595                 |
| A-B: | 56 | 400                    | 400.68             | 0.68      | 3.8381                 |
| B+C: | 56 | 260                    | 262.93             | 2.93      | 3.0263                 |
| B-C: | 56 | 140                    | 142.50             | 2.50      | 2.1320                 |

s.d.(AB) S(between runs): 3.34

Sw(within run): 2.71

S/Sw: 1.2

s.d.(BC) S(between runs): 1.85

Sw(within run): 1.51

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|     |   |     |         |
|-----|---|-----|---------|
| 820 | - | 780 | for A+B |
| 415 | - | 385 | for A-B |
| 270 | - | 250 | for B+C |
| 146 | - | 134 | for B-C |

## DUPLICATES:

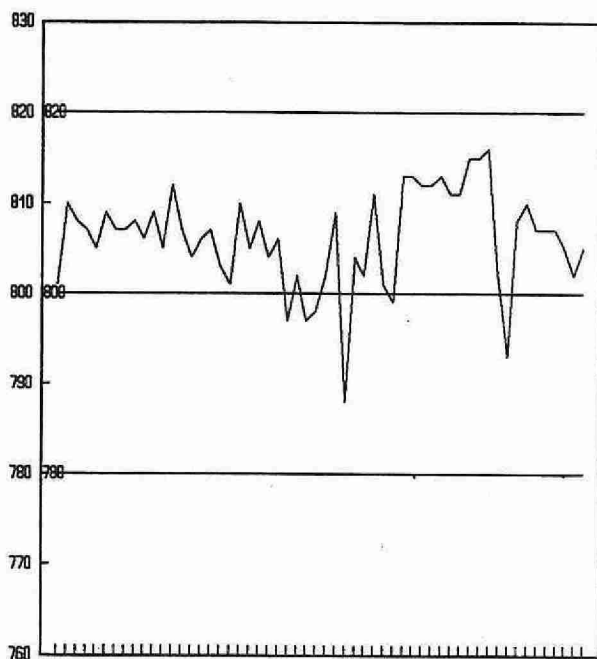
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 83           | 0.0 - 100                 | 2.4794                 | 4.7                         |
| 52           | 101 - 200                 | 1.4692                 | 1.0                         |
| 25           | 201 - 500                 | 2.5556                 | 0.8                         |
| 2            | 501 - 1000                | N.A.                   | N.A.                        |
| 162          | Overall                   | 1.9969                 |                             |

## OTHER CHECKS:

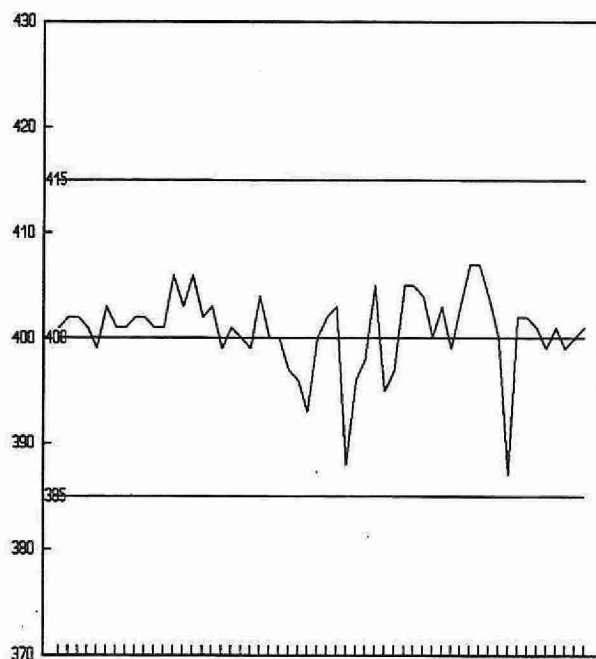
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 56 | 0.804 | 0.9802                 |

**ALUMINUM, TOTAL** ( $\mu\text{g/L}$  as Al)

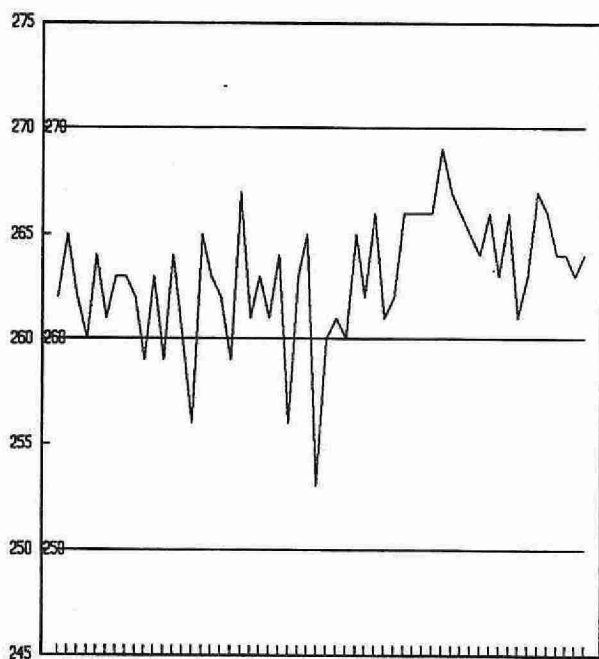
QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94



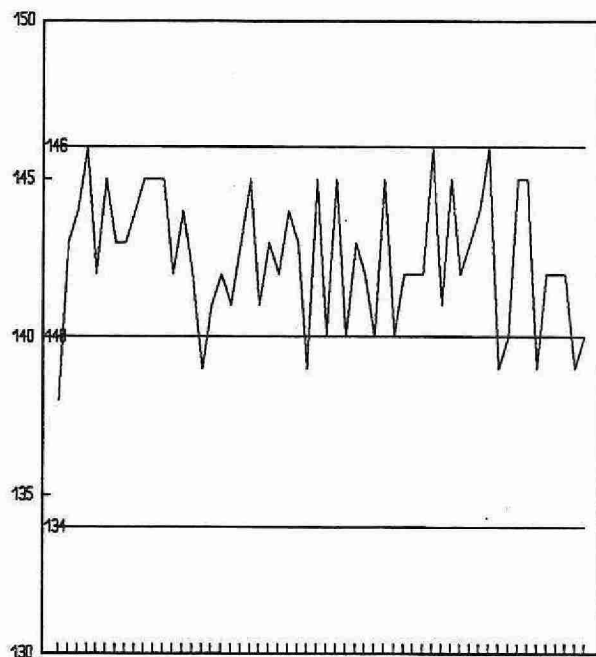
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## CADMIUM, TOTAL

### IDENTIFICATION:

|                      |                                |                   |            |
|----------------------|--------------------------------|-------------------|------------|
| Laboratory Unit      | Dorset                         | Method Introduced | 26/11/84   |
| LIS Test Name Code   | CDUT                           | Units             | µg/L as Cd |
| Work Station Code    | DOTRACE                        | Unit Code         | 063848     |
| Method Code          | 005AF2                         | Supervisor        | J. McBride |
| Method Reference No. | E3376A                         |                   |            |
| Sample Type/Matrix   | Streams, Lakes, Precipitation, |                   |            |

### SAMPLING:

|                   |  |
|-------------------|--|
| Quantity Required | 5 mL   |
| Container         | Glass or plastic, capped, acidified to 0.25% with HNO <sub>3</sub> |

### ANALYTICAL PROCEDURE:

Samples are analyzed by GFAAS at 228.8 nm.  
Approximate absorbance: 0.400 at the full scale level

### INSTRUMENTATION:

Varian graphite furnace atomic absorption spectrometer with automated sampler.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.001 | Current T value: 0.005 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 5 standards daily

### CONTROLS:

|             |                            |
|-------------|----------------------------|
| Calibration | 1 NRC sample, 3 duplicates |
| Drift       | 1 blank plus 1 standard    |

# CADMIUM, TOTAL

QUALITY CONTROL DATA FROM 19/01/94 TO 20/12/94

Laboratory Unit: Dorset

Full Scale: to 5 µg/L as Cd

## CALIBRATION CONTROL:

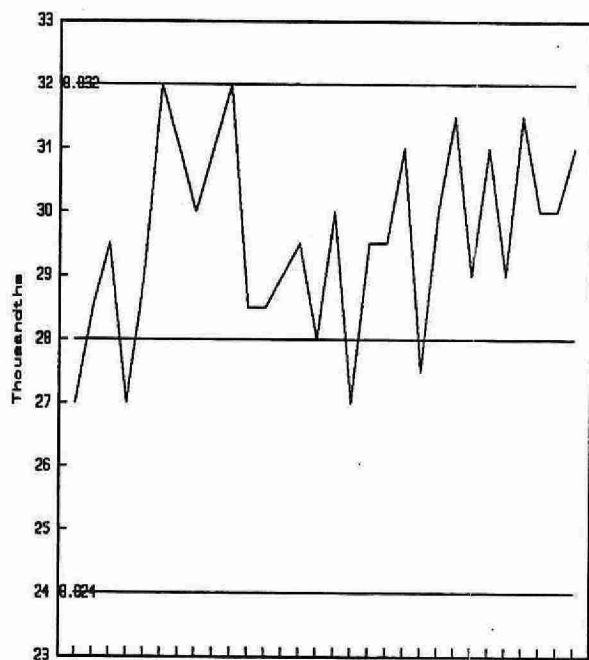
|     | n  | Mean<br>Concentration | Standard<br>Deviation (1) |
|-----|----|-----------------------|---------------------------|
| NRC | 30 | 0.0296                | 0.0015                    |

## DUPLICATES:

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 109             | 0.00 - 0.50                  | 0.0070                    | 11.3                           |
| 0               | 0.51 - 1.00                  | N.A.                      | N.A.                           |
| 0               | 1.01 - 2.50                  | N.A.                      | N.A.                           |
| 0               | 2.51 - 5.00                  | N.A.                      | N.A.                           |
| 109             | Overall                      | 0.0070                    |                                |

# CADMIUM, TOTAL ( $\mu\text{g/L}$ )

QUALITY CONTROL DATA FROM 19/01/94 TO 20/12/94



NRC REFERENCE SAMPLE

CONTROL LIMIT

## CALCIUM

### IDENTIFICATION:

|                      |                   |                   |            |
|----------------------|-------------------|-------------------|------------|
| Laboratory Unit      | Atomic Absorption | Method Introduced | 18/05/79   |
| Method Reference No. | E3146A            | Units             | mg/L as Ca |
| LIMS Product Code    | CAT3146           | Supervisor        | J. McBride |
| Sample Type/Matrix   | Precipitation     |                   |            |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 5 mL    |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.  
Approximate absorbance: 0.2 at the full scale level.

### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.005 | Current T value: 0.025 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 2 standards, e.g., QCA       |
| Drift       | BL, reslope standard every 10 samples. |



# CALCIUM

QUALITY CONTROL DATA FROM 11/01/94 TO 15/12/94

Laboratory Unit: Atomic Absorption

Full Scale: to 2.0 mg/L as Ca

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 31 | 1.20                      | 1.2046                | 0.0046    | 0.0117                    |
| B:   | 31 | 0.20                      | 0.1984                | -0.0016   | 0.0063                    |
| A+B: | 31 | 1.40                      | 1.4030                | 0.0030    | 0.0163                    |
| A-B: | 31 | 1.00                      | 1.0062                | 0.0062    | 0.0095                    |

s.d.(AB) S(between runs): 0.0094

Sw(within run): 0.0067

S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

1.35 - 1.45 for A+B  
0.96 - 1.04 for A-B

## DUPLICATES:

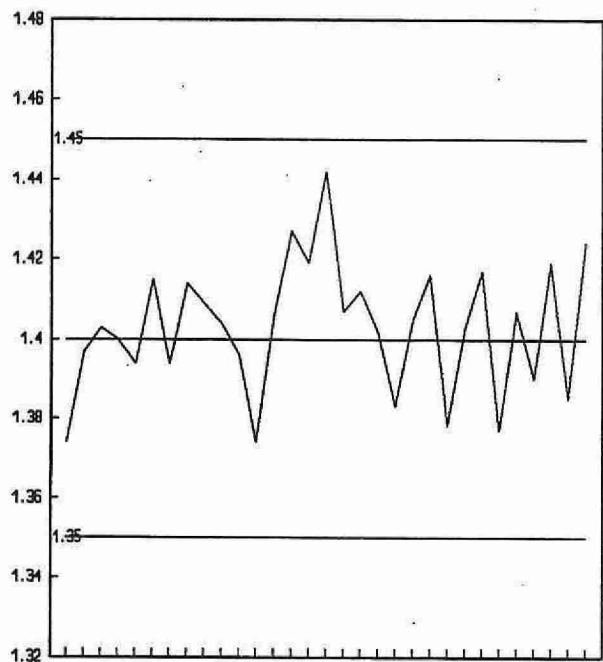
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 39              | 0.000 - 0.200                | 0.0034                    | 5.4                            |
| 12              | 0.201 - 0.400                | 0.0037                    | 1.5                            |
| 7               | 0.401 - 1.00                 | 0.0089                    | 1.3                            |
| 11              | 1.001 - 2.00                 | 0.0126                    | 0.8                            |
| 69              | OVERALL                      | 0.0059                    |                                |

## OTHER CHECKS:

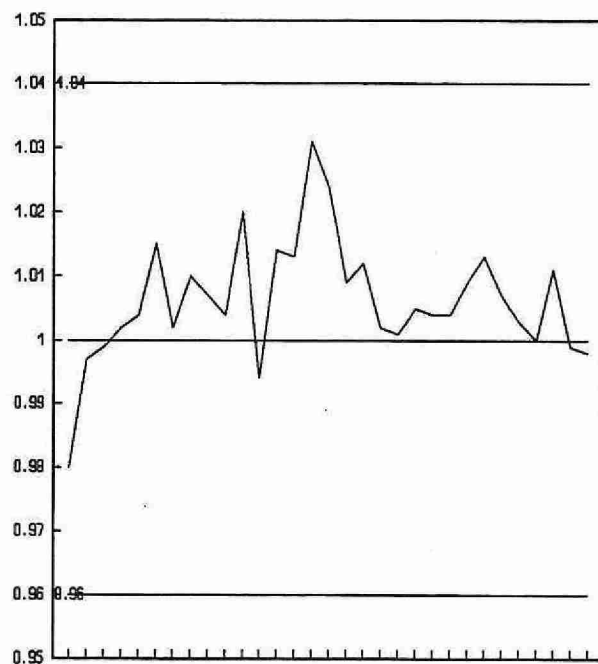
|                 | n  | Mean  | Standard<br>Deviation (1) |
|-----------------|----|-------|---------------------------|
| Long Term Blank | 31 | 0.002 | 0.0193                    |

**CALCIUM** (mg/L as Ca)

QUALITY CONTROL DATA FROM 11/01/94 TO 15/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## CALCIUM

### IDENTIFICATION:

|                      |                                      |                   |            |
|----------------------|--------------------------------------|-------------------|------------|
| Laboratory Unit      | Atomic Absorption                    | Method Introduced | 01/04/74   |
| Method Reference No. | E3171A                               | Units             | mg/L as Ca |
| LIMS Product Code    | CAT3171,CA3171,HARD3171              | Supervisor        | J. McBride |
| Sample Type/Matrix   | Surface Waters, DWSP Drinking Waters |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.14 at the full scale level.

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 11 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA                   |
| Drift       | BL every 10 samples; 2 standards every 20 samples. |

### NOTES:

Control Limits were exceeded on July 15 and August 9, 1994.

# CALCIUM

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94

Laboratory Unit: Atomic Absorption

Full Scale: to 40.0 mg/L as Ca

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 166 | 32.0                   | 31.9               | -0.1      | 0.2396                 |
| B:   | 166 | 8.00                   | 7.98               | -0.02     | 0.1225                 |
| C:   | 166 | 2.00                   | 2.00               | -0.2      | 0.2941                 |
| A+B: | 166 | 40.0                   | 39.8               | -0.1      | 0.2416                 |
| A-B: | 166 | 24.0                   | 23.9               | 0.002     | 0.0383                 |
| B+C: | 166 | 10.0                   | 9.98               | -0.02     | 0.1388                 |
| B-C: | 166 | 6.00                   | 5.98               | -0.02     | 0.1170                 |

s.d.(AB) S(between runs): 0.19

Sw(within run): 0.17

S/Sw: 1.1

s.d.(BC) S(between runs): 0.09

Sw(within run): 0.08

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 38.8 | - | 41.2 | for | A+B |
| 23.1 | - | 24.9 | for | A-B |
| 9.60 | - | 10.4 | for | B+C |
| 5.70 | - | 6.30 | for | B-C |

## DUPLICATES:

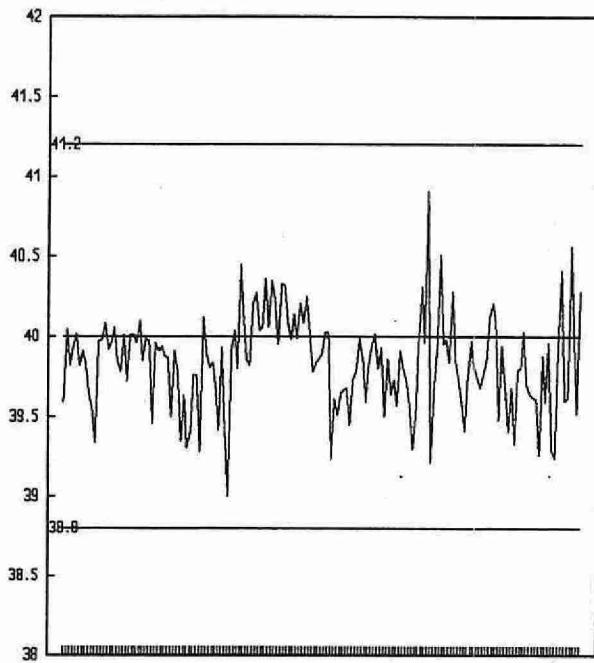
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 40           | 0.00 - 4.00               | 0.0430                 | 1.9                         |
| 43           | 4.01 - 8.00               | 0.0850                 | 1.2                         |
| 103          | 8.01 - 20.0               | 0.1228                 | 1.4                         |
| 188          | 20.1 - 40.0               | 0.1774                 | 0.9                         |
| 374          | Overall                   |                        |                             |

## OTHER CHECKS:

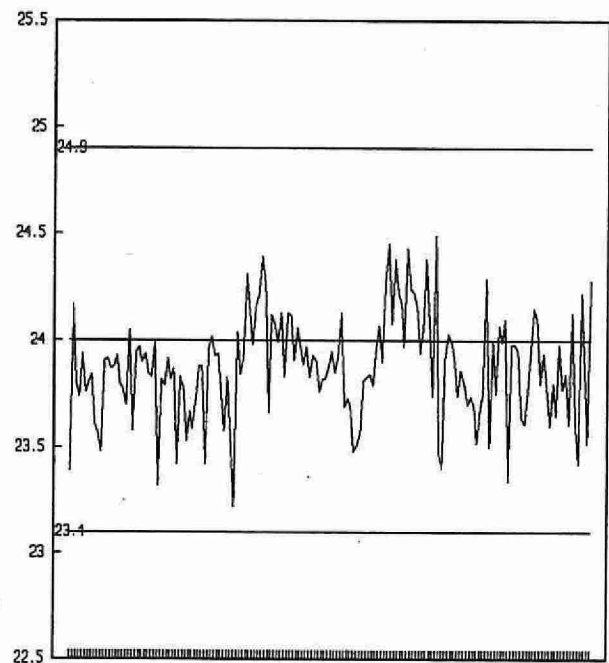
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 166 | -0.008 | 0.0345                 |

CALCIUM (mg/L as Ca)

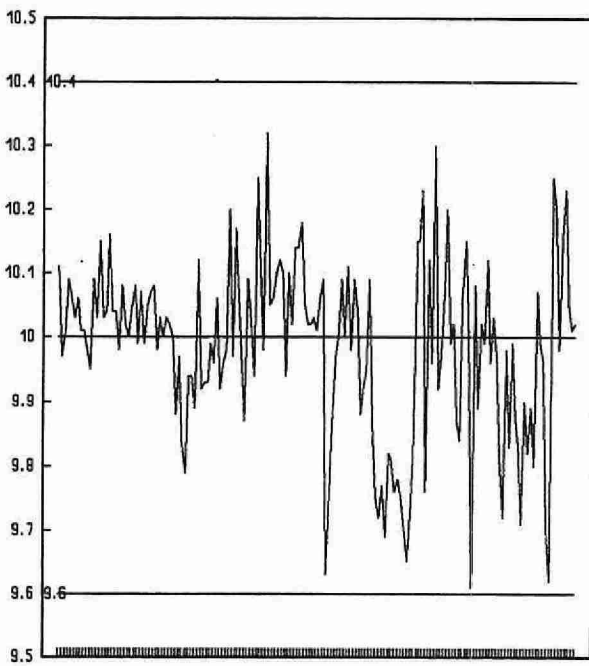
QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94



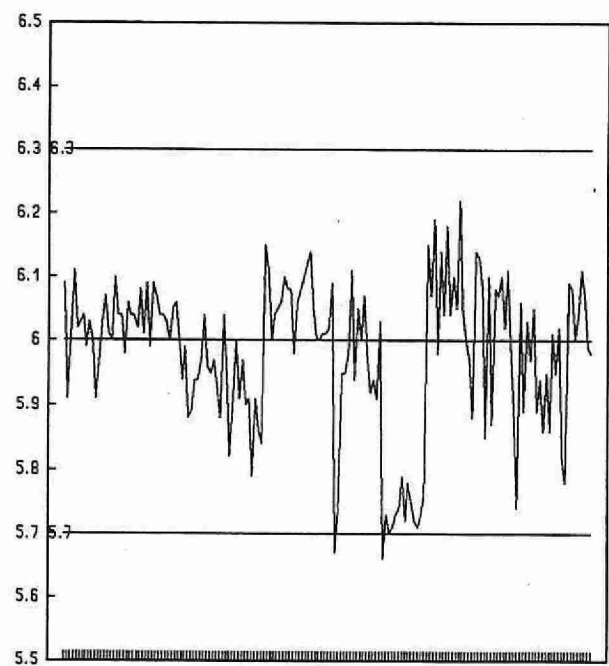
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## CALCIUM

### IDENTIFICATION:

|                      |   |                   |            |
|----------------------|---|-------------------|------------|
| Laboratory Unit      | Atomic Absorption   | Method Introduced | 08/04/86   |
| Method Reference No. | E3217A  | Units             | mg/L as Ca |
| LIMS Product Code    | CAT3217,CATS3217,HARD3217                                       | Supervisor        | J. McBride |
| Sample Type/Matrix   | Domestic Waters, Leachates, Effluents, Sewage,Industrial Wastes |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.17 at the full scale level.

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

BL plus 11 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA                   |
| Drift       | BL every 10 samples; 2 standards every 20 samples. |

# CALCIUM

QUALITY CONTROL DATA FROM 04/01/94 TO 29/12/94

Laboratory Unit: Absorption

Full Scale: to 200.0 mg/L as Ca

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 115 | 160.0                  | 159.13             | -0.87     | 1.9236                 |
| B:   | 115 | 40.0                   | 40.13              | 0.13      | 0.9601                 |
| C:   | 115 | 10.0                   | 10.02              | 0.02      | 0.3684                 |
| A+B: | 115 | 200.0                  | 199.19             | -0.81     | 2.6257                 |
| A-B: | 115 | 120.0                  | 118.93             | -1.07     | 1.7345                 |
| B+C: | 115 | 50.0                   | 50.14              | 0.14      | 1.2136                 |
| B-C: | 115 | 30.0                   | 30.11              | 0.11      | 0.8612                 |

s.d.(AB) S(between runs): 1.52

Sw(within run): 1.23

S/Sw: 1.2

s.d.(BC) S(between runs): 0.73

Sw(within run): 0.61

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 190  | - | 210  | for | A+B |
| 113  | - | 127  | for | A-B |
| 44.5 | - | 54.5 | for | B+C |
| 27.0 | - | 33.0 | for | B-C |

## DUPLICATES:

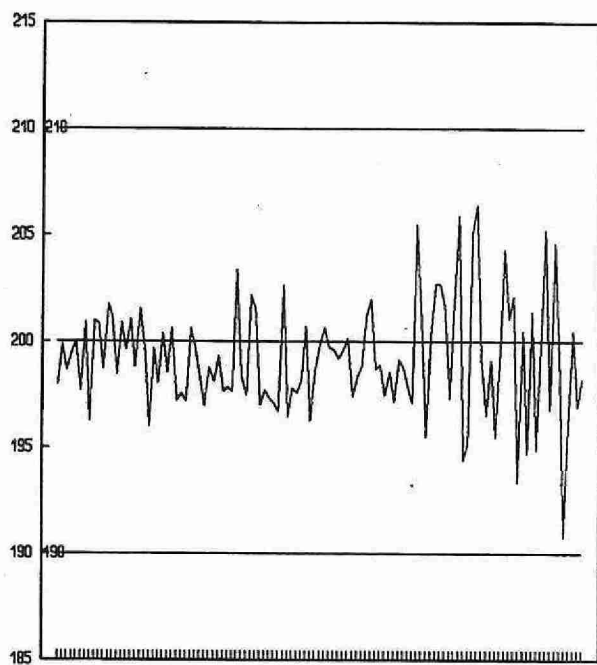
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 45           | 0.00 - 20.00              | 0.3428                 | 4.0                         |
| 54           | 20.01 - 40.00             | 0.7640                 | 2.3                         |
| 130          | 40.01 - 100.00            | 1.4370                 | 1.9                         |
| 52           | 100.01 - 200.00           | 2.1064                 | 1.6                         |
| 281          | Overall                   | 1.2410                 |                             |

## OTHER CHECKS:

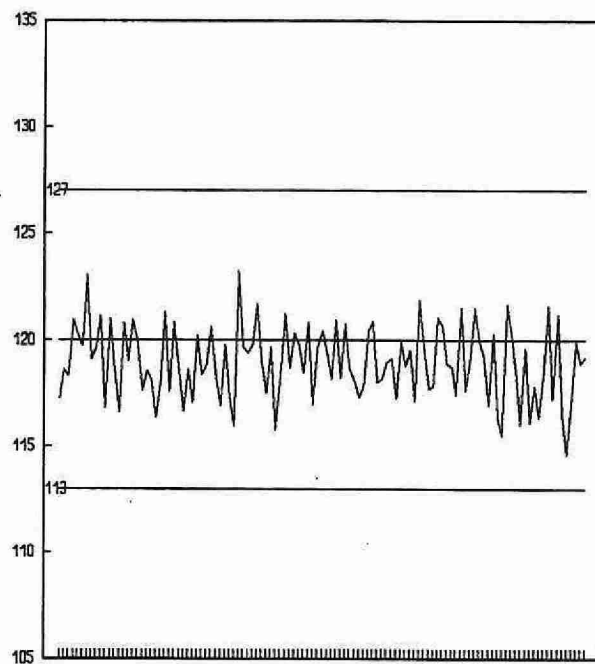
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 115 | -0.379 | 0.3989                 |

# CALCIUM (mg/L as Ca)

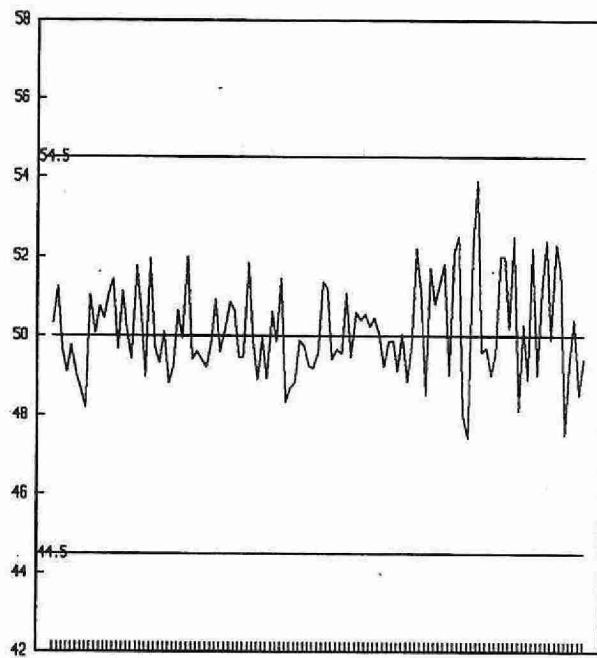
QUALITY CONTROL DATA FROM 04/01/94 TO 29/12/94



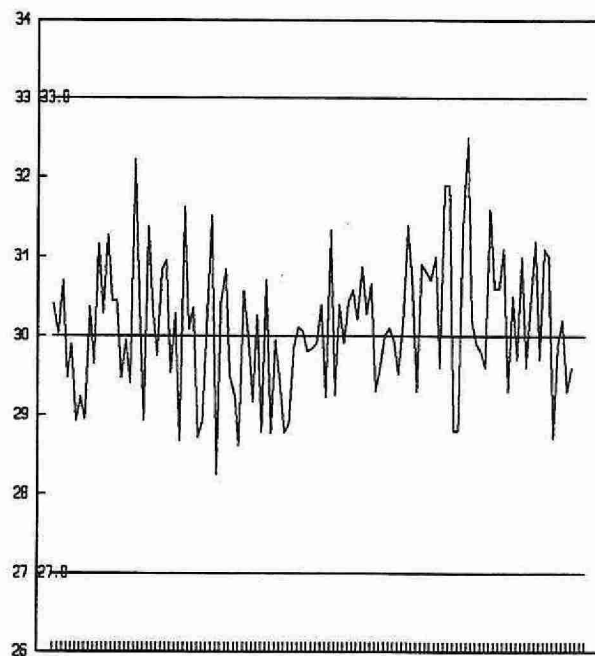
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT



## CALCIUM

### IDENTIFICATION:

|                      |                |                   |            |
|----------------------|----------------|-------------------|------------|
| Laboratory Unit      | Dorset         | Method Introduced | 20/07/88   |
| LIS Test Name Code   | CAUR           | Units             | mg/L as Ca |
| Work Station Code    | DOFLAME        | Unit Code         | 064820     |
| Method Code          | 002CA1         | Supervisor        | J. McBride |
| Method Reference No. | E3249A         |                   |            |
| Sample Type/Matrix   | Rivers, Lakes, |                   |            |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 5 mL    |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.2 at the full scale level.

### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                       |                      |
|--------------------------------|-----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.1 |
|--------------------------------|-----------------------|----------------------|

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA       |
| Drift       | BL, reslope standard every 10 samples. |

### NOTES:

The control standards are corrected for the LTB from which they were made.

# CALCIUM

QUALITY CONTROL DATA FROM 13/01/94 TO 22/12/94

Laboratory Unit: Dorset

Full Scale: to 8.0 mg/L as Ca

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 80 | 6.4                    | 6.414              | 0.014     | 0.0347                 |
| B:   | 80 | 1.6                    | 1.607              | 0.007     | 0.0145                 |
| C:   | 80 | 0.4                    | 0.407              | 0.007     | 0.0055                 |
| A+B: | 80 | 8.0                    | 8.009              | 0.009     | 0.0362                 |
| A-B: | 80 | 4.8                    | 4.807              | 0.007     | 0.0372                 |
| B+C: | 80 | 2.0                    | 2.003              | 0.003     | 0.0177                 |
| B-C: | 80 | 1.2                    | 1.200              | 0.0003    | 0.0139                 |

s.d.(AB) S(between runs): 0.03

Sw(within run): 0.03

S/Sw: 1.0

s.d.(BC) S(between runs): 0.01

Sw(within run): 0.01

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 7.85 | - | 8.15 | for | A+B |
| 4.69 | - | 4.91 | for | A-B |
| 1.95 | - | 2.05 | for | B+C |
| 1.16 | - | 1.24 | for | B-C |

## DUPLICATES:

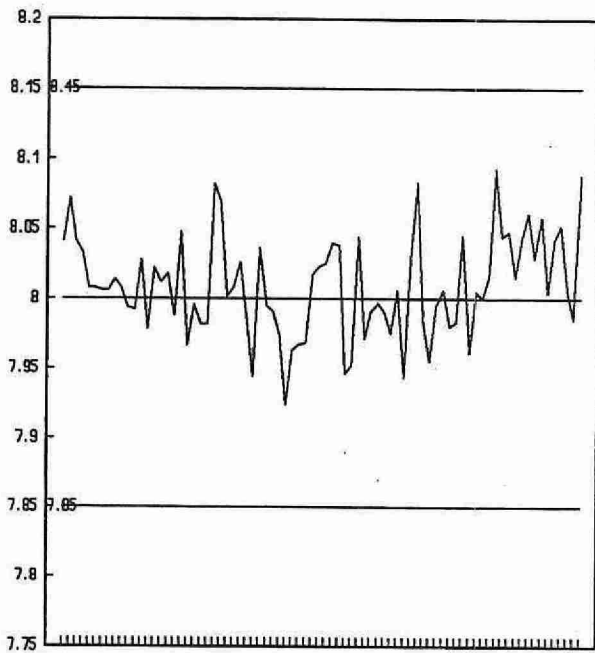
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 46           | 0.00 - 0.80               | 0.0090                 | 6.8                         |
| 16           | 0.81 - 1.60               | 0.0821                 | 6.0                         |
| 63           | 1.61 - 4.00               | 0.0565                 | 3.4                         |
| 0            | 4.01 - 8.00               | N.A.                   | N.A.                        |
| 125          | Overall                   | 0.0359                 |                             |

## OTHER CHECKS:

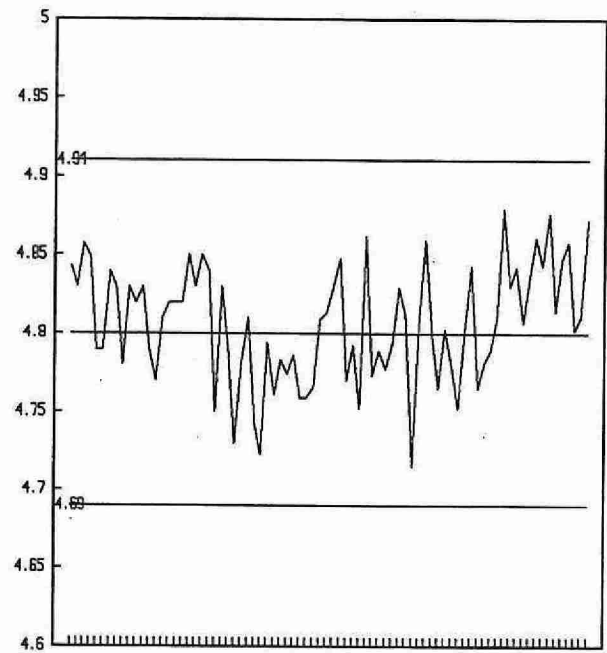
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 80 | 0.006 | 0.0062                 |

**CALCIUM** (mg/L as Ca)

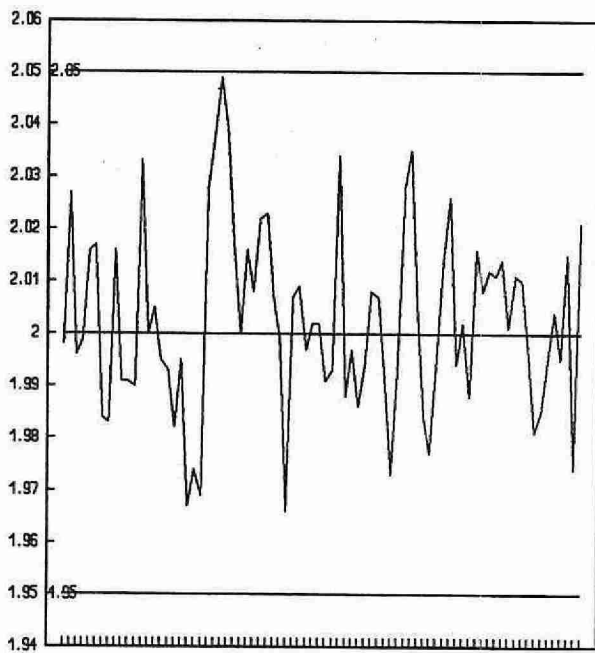
QUALITY CONTROL DATA FROM 13/01/94 TO 22/12/94



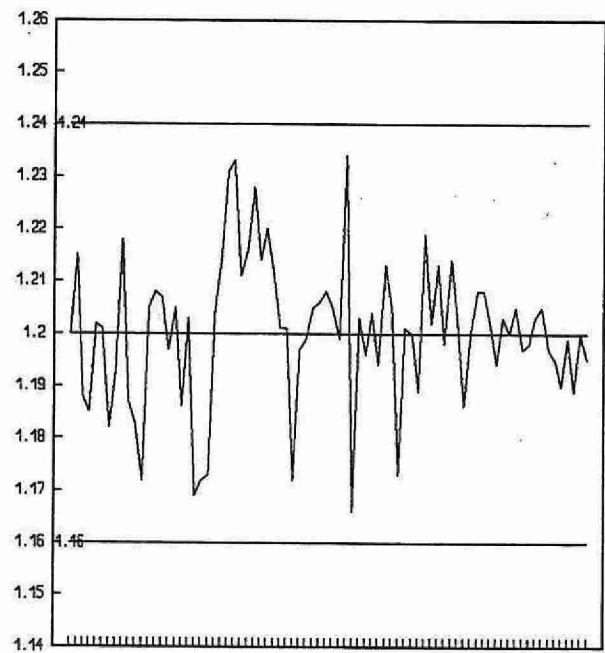
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## CARBON, DISSOLVED INORGANIC

### IDENTIFICATION:

|                      |  |                   |            |
|----------------------|--|-------------------|------------|
| Laboratory Unit      | Dorset   | Method Introduced | 03/06/80   |
| LIS Test Name Code   | DIC  | Units             | mg/L as C  |
| Work Station Code    | DODIC  | Unit Code         | 064806     |
| Method Code          | 1127C2   | Supervisor        | J. McBride |
| Method Reference No. | E3028A   |                   |            |
| Sample Type/Matrix   | Streams, Lakes, Groundwater and Soil Leachates |                   |            |

### SAMPLING:

|                   |       |
|-------------------|-------|
| Quantity Required | 50 mL |
| Container         | Glass |

### ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: air (CO<sub>2</sub>-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Two analytical ranges are obtained from the output of the colourimeter.

### REPORTING:

|                                |                       |                      |
|--------------------------------|-----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.1 |
|--------------------------------|-----------------------|----------------------|

### CALIBRATION:

BL plus 9 standards daily

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTB plus 3 standards, e.g. QCA, QCB, QCC                       |
| Drift       | BL every 10 samples; BL plus 1 check standard every 20 samples |

# CARBON, DISSOLVED INORGANIC

QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94

Laboratory : Dorset

Full Scale: to 10.0 mg/L as C

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 65 | 8.0                    | 7.89               | -0.11     | 0.1408                 |
| B:   | 65 | 4.0                    | 3.90               | -0.10     | 0.0911                 |
| C:   | 65 | 0.8                    | 0.74               | -0.06     | 0.0433                 |
| A+B: | 65 | 12.0                   | 11.79              | -0.21     | 0.2042                 |
| A-B: | 65 | 4.0                    | 3.99               | -0.01     | 0.1205                 |
| B+C: | 65 | 4.8                    | 4.64               | -0.16     | 0.1192                 |
| B-C: | 65 | 3.2                    | 3.16               | -0.04     | 0.0782                 |

s.d.(AB) S(between runs): 0.12

Sw(within run): 0.09

S/Sw: 1.4

s.d.(BC) S(between runs): 0.07

Sw(within run): 0.06

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 11.4 | - | 12.6 | for | A+B |
| 3.6  | - | 4.4  | for | A-B |
| 4.4  | - | 5.2  | for | B+C |
| 2.9  | - | 3.5  | for | B-C |

## DUPLICATES:

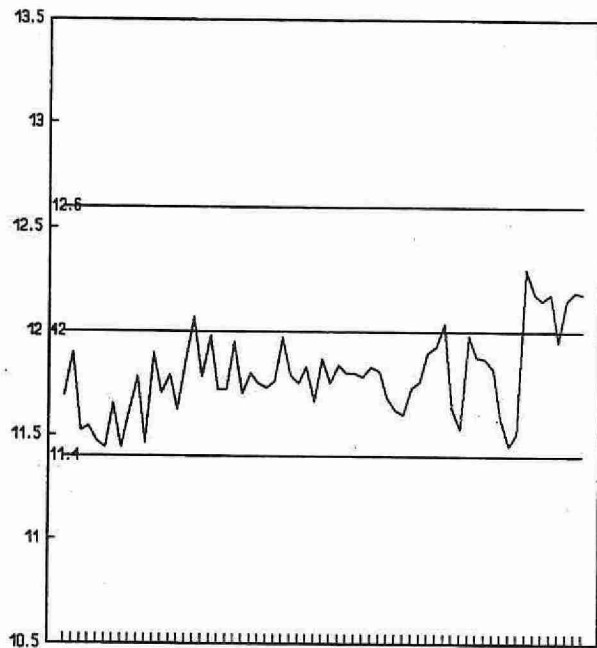
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 57           | 0.00 - 1.00               | 0.0213                 | 3.5                         |
| 68           | 1.01 - 2.00               | 0.0451                 | 3.3                         |
| 53           | 2.01 - 5.00               | 0.0747                 | 3.2                         |
| 16           | 5.01 - 10.0               | 0.0986                 | 2.1                         |
| 194          | Overall                   | 0.0452                 |                             |

## OTHER CHECKS:

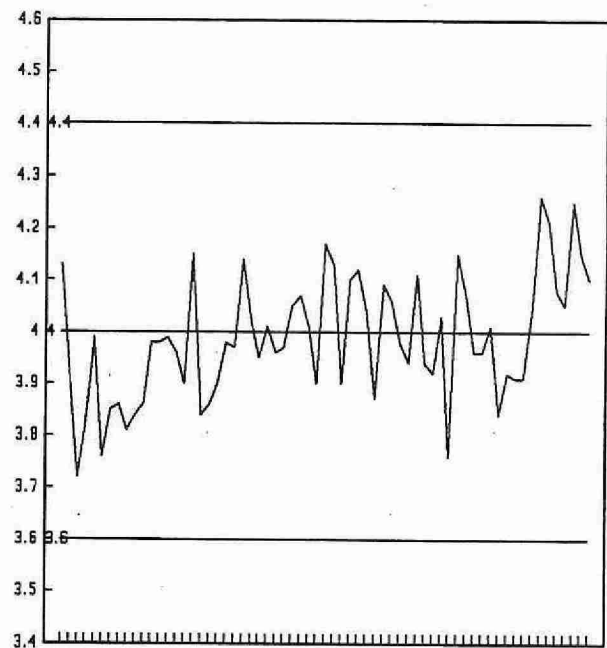
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 65 | 0.2015 | 0.0450                 |

**CARBON, DISSOLVED INORGANIC** (mg/L as C)

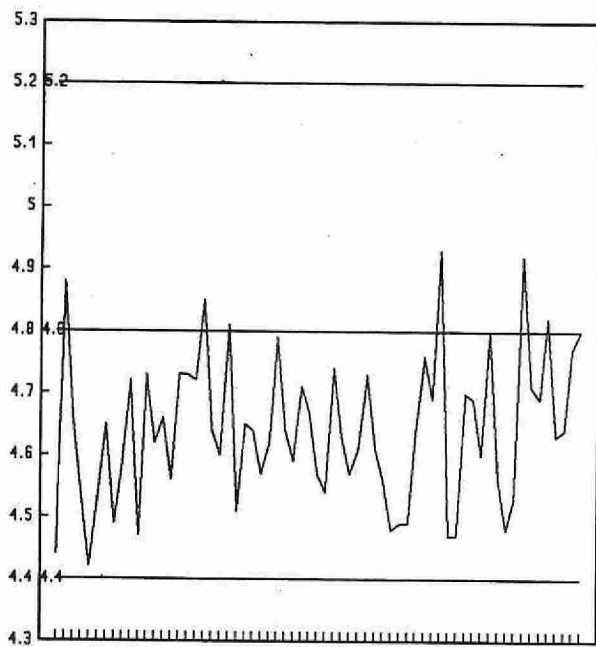
QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94



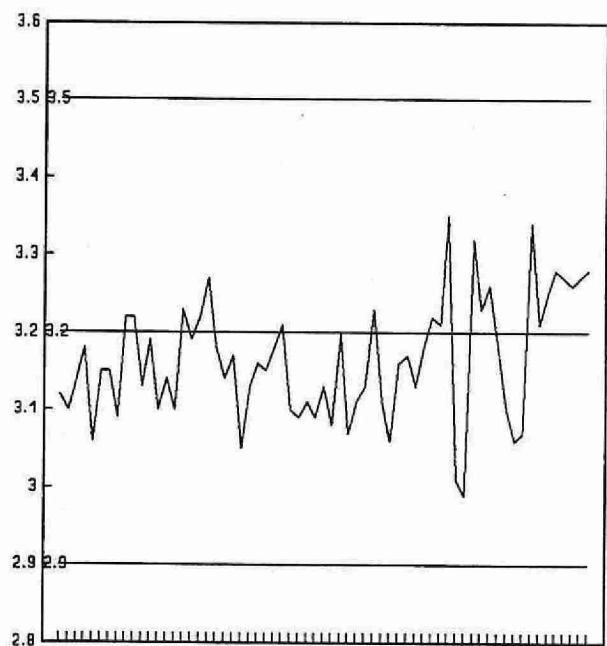
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## CARBON, DISSOLVED INORGANIC

### IDENTIFICATION:

|                      |  |                   |             |
|----------------------|--|-------------------|-------------|
| Laboratory Unit      | Colourimetry   | Method Introduced | 01/04/78    |
| Method Reference No. | E3370A   | Units             | mg/L as C   |
| LIMS Product Code    | DCSI3370   | Supervisor        | M. Rawlings |
| Sample Type/Matrix   | Rivers, Lakes, Precipitation, Soil Extracts, Effluents, Domestic Water Supplies, Leachates, Sewages, Industrial Wastes |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

Dissolved organic carbon, and reactive silicates are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: air (CO<sub>2</sub>-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1.0 |
|--------------------------------|----------------------|----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 standards, e.g., QCA                |
| Drift       | BL every 10 samples; standard every 20 samples. |

### NOTES:

Sept.'94 the method codes ROM-E3176A, and E3178A were amalgamated and a new method code ROM-E3370A was generated.

Nov'94 the full scale range was changed to 80 mg/L as C.

# CARBON, DISSOLVED INORGANIC

QUALITY CONTROL DATA FROM 04/01/94 TO 27/10/94

Laboratory Unit: Colourimetry

Full Scale: to 40.0 mg/L as C

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 120 | 32.0                   | 31.97              | -0.03     | 0.5377                 |
| B:   | 120 | 8.00                   | 7.98               | -0.02     | 0.2193                 |
| C:   | 120 | 2.00                   | 2.02               | 0.02      | 0.1376                 |
| A+B: | 120 | 40.0                   | 39.94              | -0.06     | 0.6611                 |
| A-B: | 120 | 24.0                   | 23.993             | -0.007    | 0.4871                 |
| B+C: | 120 | 10.0                   | 9.99               | -0.01     | 0.3131                 |
| B-C: | 120 | 6.00                   | 5.96               | -0.04     | 0.1898                 |

s.d.(AB) S(between runs): 0.41

Sw(within run): 0.34

S/Sw: 1.2

s.d.(BC) S(between runs): 0.18

Sw(within run): 0.41

S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 37.8 | - | 42.2 | for | A+B |
| 22.5 | - | 25.5 | for | A-B |
| 8.90 | - | 11.1 | for | B+C |
| 5.30 | - | 6.70 | for | B-C |

## DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 82           | 0.00 - 4.00               | 0.2873                 | 32.2                        |
| 22           | 4.01 - 8.00               | 0.3675                 | 9.5                         |
| 49           | 8.01 - 20.0               | 0.4855                 | 5.6                         |
| 207          | 20.1 - 40.0               | 0.6825                 | 2.5                         |
| 360          | Overall                   | 0.5316                 |                             |

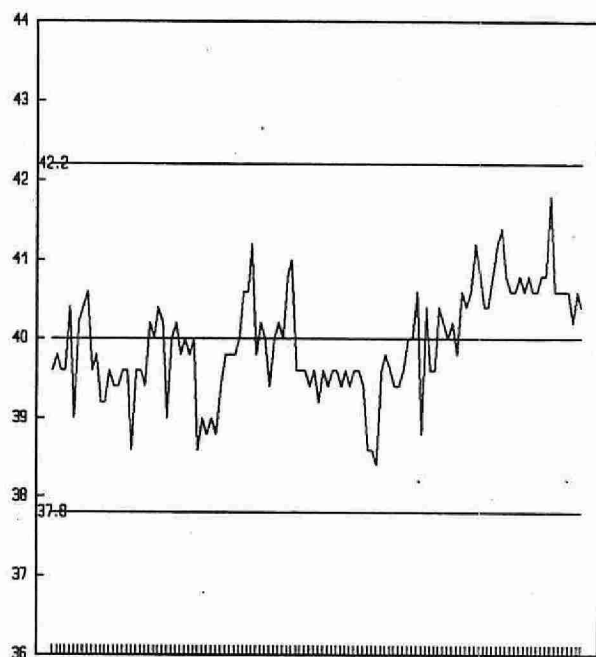
## OTHER CHECKS:

|                 | n   | Mean    | Standard Deviation (1) |
|-----------------|-----|---------|------------------------|
| Long Term Blank | 120 | -0.0383 | 0.1403                 |

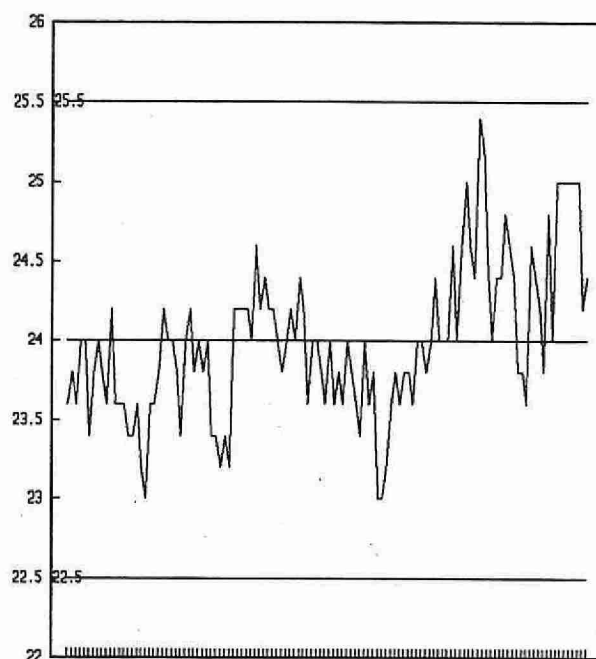


CARBON, DISSOLVED INORGANIC (mg/L as C)

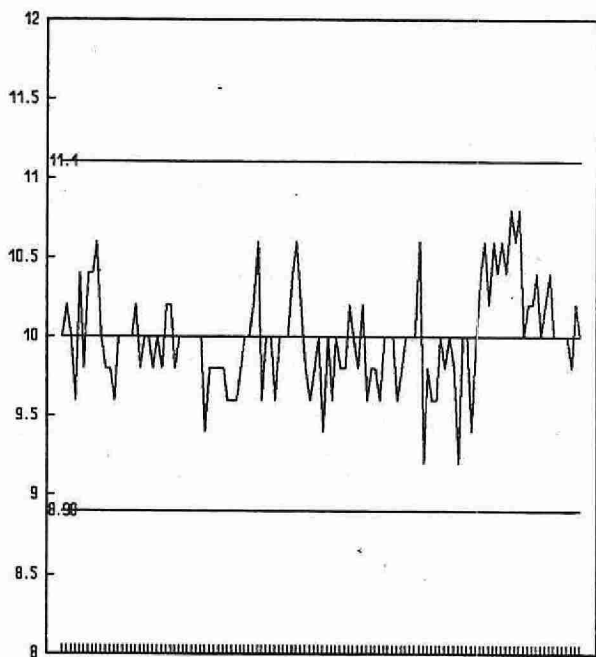
QUALITY CONTROL DATA FROM 04/01/94 TO 27/10/94



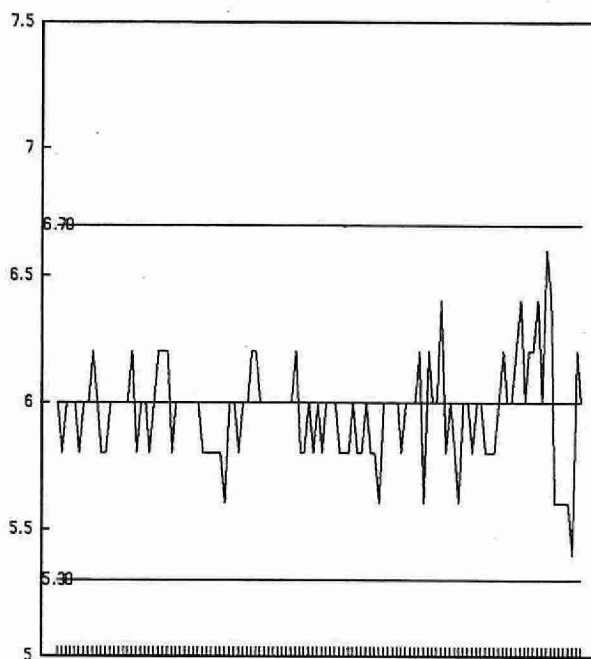
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

# CARBON, DISSOLVED INORGANIC

QUALITY CONTROL DATA FROM 31/10/94 TO 22/12/94

Laboratory Unit: Colourimetry

Full Scale: to 80.0 mg/L as C

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 26 | 64.0                   | 64.4               | 0.4       | 0.3295                 |
| B:   | 26 | 16.0                   | 15.7               | -0.3      | 0.2130                 |
| C:   | 26 | 4.00                   | 3.96               | -0.04     | 0.1602                 |
| A+B: | 26 | 80.0                   | 80.1               | 0.1       | 0.4077                 |
| A-B: | 26 | 48.0                   | 48.7               | 0.7       | 0.3763                 |
| B+C: | 26 | 20.0                   | 19.7               | -0.3      | 0.3302                 |
| B-C: | 26 | 12.0                   | 11.8               | -0.2      | 0.1816                 |

s.d.(AB) S(between runs): 0.28

Sw(within run): 0.27

S/Sw: 1.1

s.d.(BC) S(between runs): 0.19

Sw(within run): 0.13

S/Sw: 1.5

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 78.1 | - | 81.9 | for | A+B |
| 46.6 | - | 49.5 | for | A-B |
| 19.1 | - | 20.9 | for | B+C |
| 11.4 | - | 12.6 | for | B-C |

## DUPLICATES:

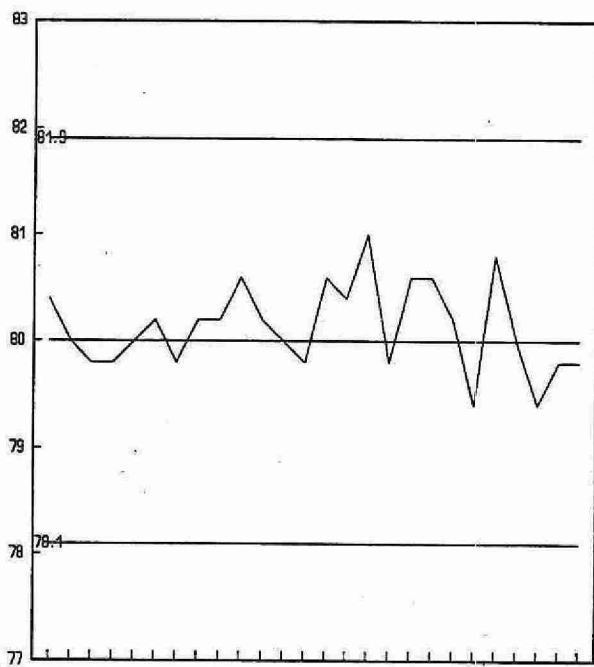
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 33           | 0.00 - 8.00               | 0.2982                 | 26.4                        |
| 0            | 8.01 - 16.0               | N.A.                   | N.A.                        |
| 23           | 16.1 - 40.0               | 0.3462                 | 1.4                         |
| 21           | 40.1 - 80.0               | 0.5691                 | 1.6                         |
| 77           | Overall                   | 0.3727                 |                             |

## OTHER CHECKS:

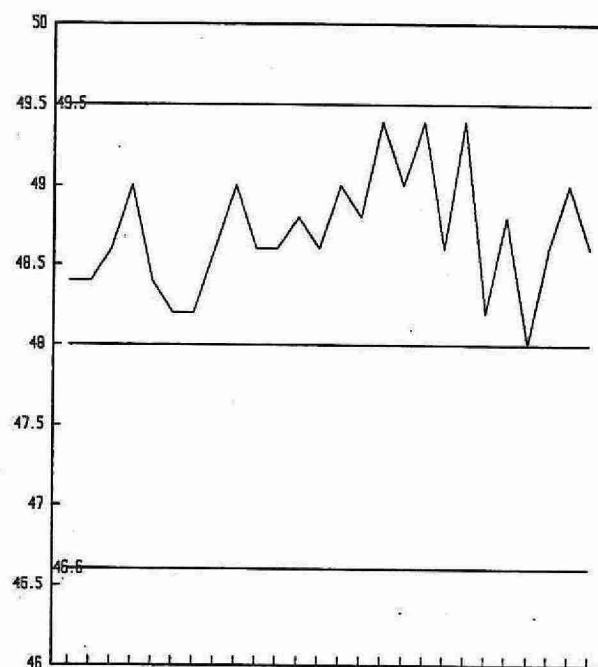
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 26 | 0.031 | 0.2241                 |

# CARBON, DISSOLVED INORGANIC (mg/L as C)

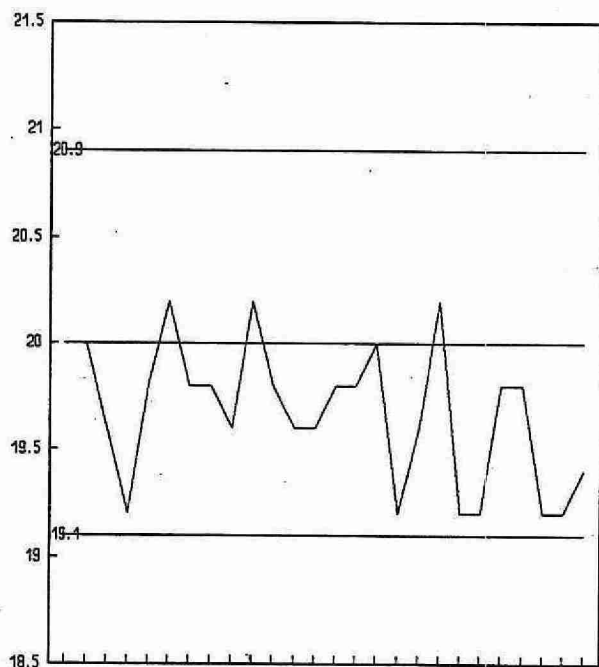
QUALITY CONTROL DATA FROM 31/10/94 TO 22/12/94



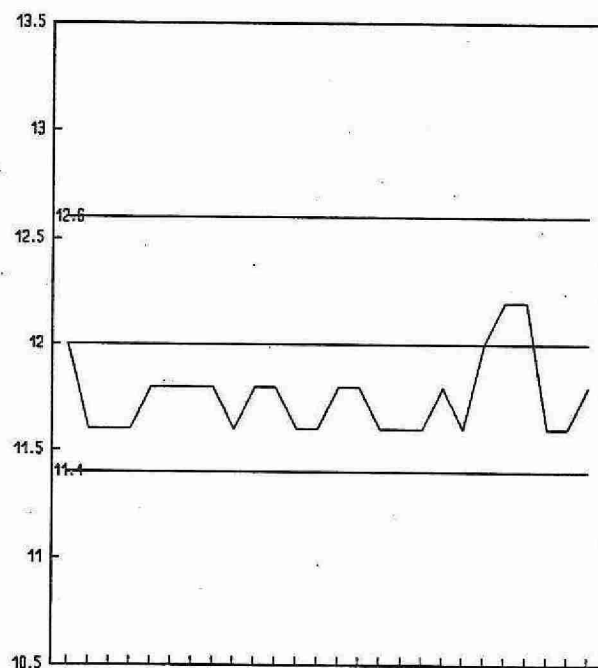
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## CARBON, DISSOLVED ORGANIC

### IDENTIFICATION:

|                      |  |                   |             |
|----------------------|--|-------------------|-------------|
| Laboratory Unit      | Colourimetry   | Method Introduced | 01/04/78    |
| Method Reference No. | E3370A   | Units             | mg/L as C   |
| LIMS Product Code    | DCSI3370   | Supervisor        | M. Rawlings |
| Sample Type/Matrix   | Rivers, Lakes, Precipitation, Soil Extracts, Effluents, Domestic Water Supplies, Leachates, Sewages, Industrial Wastes |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Using an automated system, the supernatant from a settled sample is acidified and flushed with nitrogen gas to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide gas by exposure to ultra-violet light (UV) in acid-persulphate media. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved organic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

Dissolved inorganic carbon, and reactive silicates are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: nitrogen and air (CO<sub>2</sub>-free) supplies with flow controls, dialysis unit, UV digester. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.1 | Current T value: 0.5 |
|--------------------------------|----------------------|----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA                   |
| Drift       | BL every 10 samples; 2 standards every 20 samples. |

### NOTES:

Sept.'94 the method codes ROM-E3176A, and E3178A were amalgamated and a new method code ROM-E3370A was generated.

# CARBON, DISSOLVED ORGANIC

QUALITY CONTROL DATA FROM 05/01/94 TO 23/12/94

Laboratory Unit: Colourimetry

Full Scale: to 20.0 mg/L as C

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 146 | 16.0                   | 16.001             | 0.001     | 0.1195                 |
| B:   | 146 | 4.00                   | 3.96               | -0.04     | 0.0735                 |
| C:   | 146 | 1.00                   | 1.02               | 0.02      | 0.0794                 |
| A+B: | 146 | 20.0                   | 19.96              | -0.04     | 0.1509                 |
| A-B: | 146 | 12.0                   | 12.04              | 0.04      | 0.1288                 |
| B+C: | 146 | 5.00                   | 4.99               | -0.01     | 0.1278                 |
| B-C: | 146 | 3.00                   | 2.95               | -0.05     | 0.0840                 |

s.d.(AB) S(between runs): 0.099

Sw(within run): 0.091

S/Sw: 1.1

s.d.(BC) S(between runs): 0.077

Sw(within run): 0.059

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 19.3 | - | 20.7 | for | A+B |
| 11.5 | - | 12.5 | for | A-B |
| 4.60 | - | 5.40 | for | B+C |
| 2.76 | - | 3.24 | for | B-C |

## DUPLICATES:

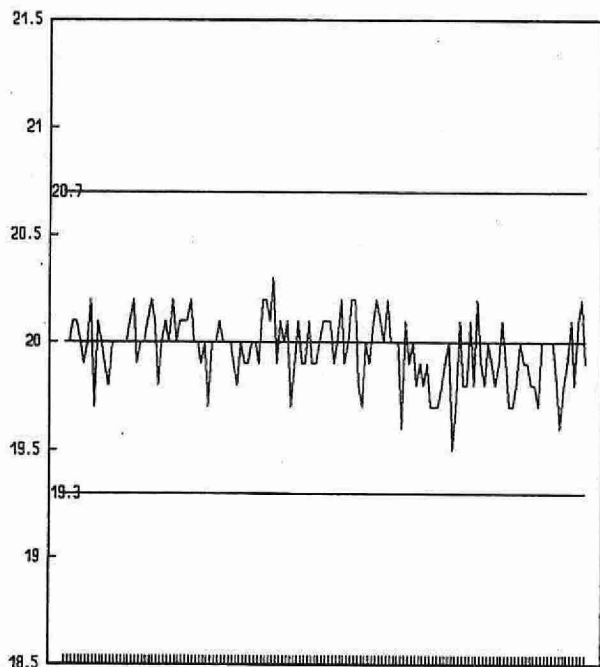
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 140          | 0.00 - 2.00               | 0.0924                 | 7.7                         |
| 121          | 2.01 - 4.00               | 0.1093                 | 7.7                         |
| 129          | 4.01 - 10.0               | 0.1362                 | 7.0                         |
| 34           | 10.1 - 20.0               | 0.1901                 | 1.7                         |
| 424          | Overall                   | 0.1157                 |                             |

## OTHER CHECKS:

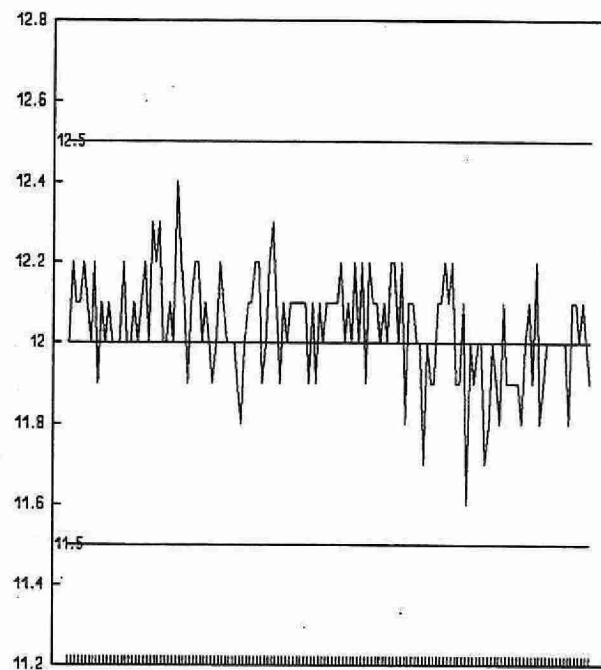
|                 | n   | Mean  | Standard Deviation (1) |
|-----------------|-----|-------|------------------------|
| Long Term Blank | 146 | 0.031 | 0.0818                 |

CARBON, DISSOLVED ORGANIC (mg/L as C)

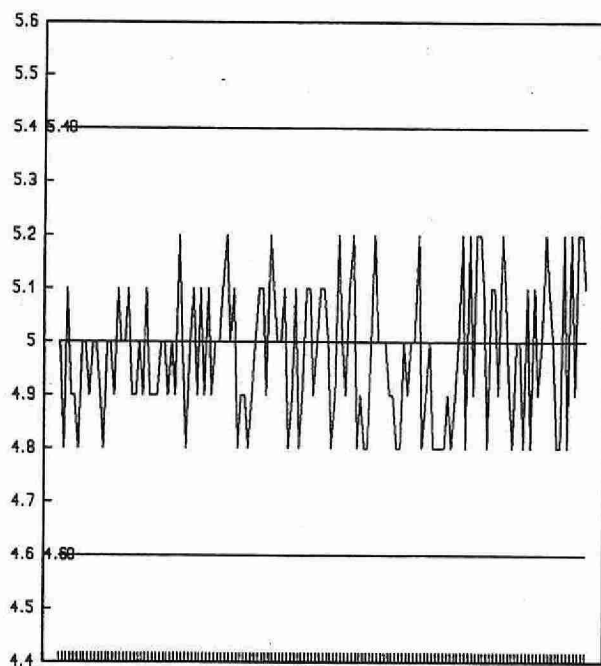
QUALITY CONTROL DATA FROM 04/01/93 TO 23/12/93



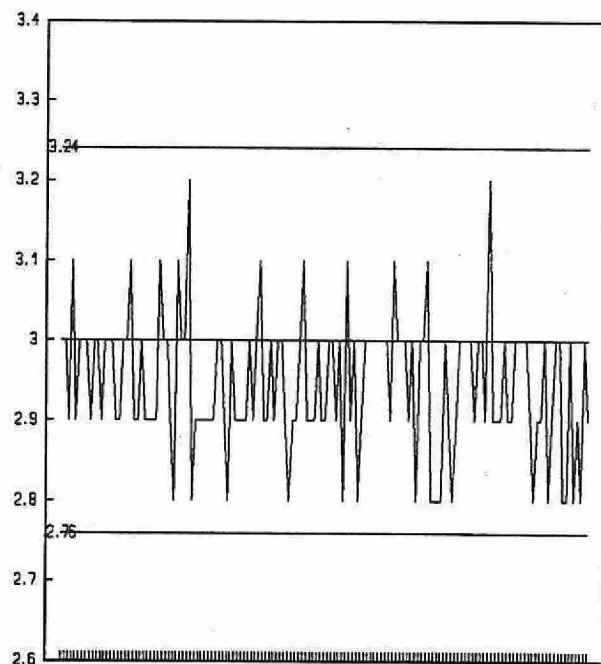
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## CARBON, TOTAL ORGANIC

### IDENTIFICATION:

|                      |                                  |                   |            |
|----------------------|----------------------------------|-------------------|------------|
| Laboratory Unit      | MISA                             | Method Introduced | 08/12/93   |
| Method Reference No. | E3247B                           | Units             | mg/L as C  |
| LIMS Product Code    | CARB3247                         | Supervisor        | J. McBride |
| Sample Type/Matrix   | Industrial Effluents, and Sewage |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 500 mL           |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

If particles in the sample are greater than about 2 mm diameter the sample is homogenized and /or pH is lowered if necessary. Automated acidification and purging are done to remove any inorganic carbon at a temperature of 820 C. An IR detector measures the CO<sub>2</sub>.

### INSTRUMENTATION:

Dorhman DC-190 Carbon Analyzer.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

A solution of potassium biphthalate is used to calibrate the instrument.

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | 2 Calibration Control Standards, eg QCA.                         |
| Blanks      | DDW  |
| Drift       | 25 ppm Check standard and a blank every 10 samples               |
| Precision   | Duplicate sample at least every 10 samples to a maximum of three |

# CARBON, TOTAL ORGANIC

QUALITY CONTROL DATA FROM 15/01/94 TO 21/12/94

Laboratory Unit: MISA

Full Scale: to 25 mg/L as C

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 74 | 20.0                   | 20.2               | 0.2       | 0.6707                 |
| B:   | 74 | 5.0                    | 5.2                | 0.2       | 0.3358                 |
| A+B: | 74 | 25.0                   | 25.4               | 0.4       | 0.8619                 |
| A-B: | 74 | 15.0                   | 14.9               | -0.1      | 0.6184                 |

s.d.(AB)

S(between runs): 0.53

Sw(within run): 0.44

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

22.9 - 27.1 for A+B  
13.4 - 16.6 for A-B

## DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 54           | 0.0 - 2.5                 | 0.1469                 | 9.9                         |
| 51           | 2.6 - 5.0                 | 0.2583                 | 8.4                         |
| 61           | 5.1 - 12.5                | 0.4360                 | 6.5                         |
| 21           | 12.6 - 25.0               | 0.5422                 | 4.0                         |
| 187          | Overall                   | 0.3143                 |                             |

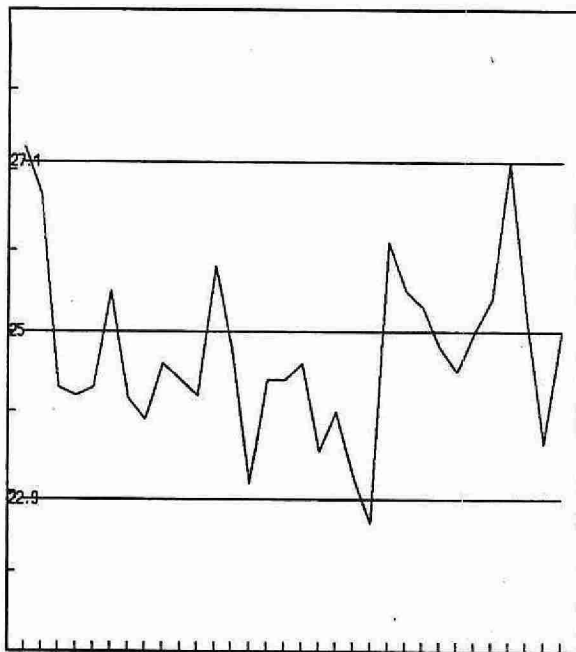
## OTHER CHECKS:

|              | n  | Mean    | Standard Deviation (1) |
|--------------|----|---------|------------------------|
| Method Blank | 74 | -0.1023 | 0.4009                 |

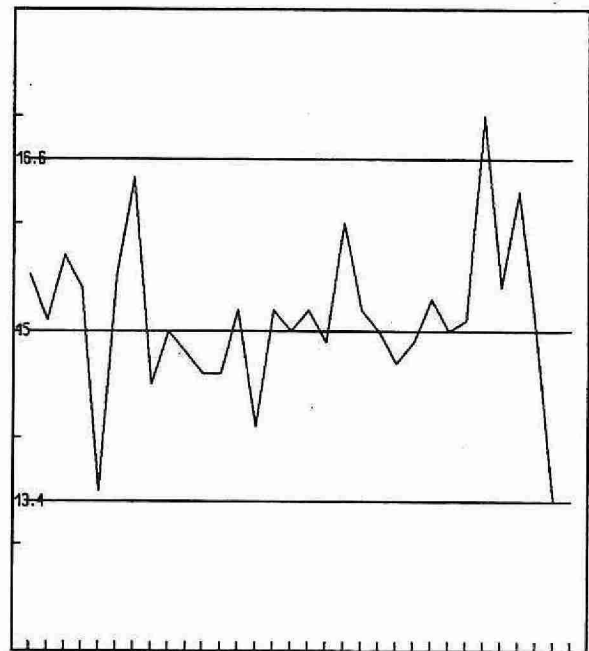


CARBON, TOTAL ORGANIC (mg/L as C)

QUALITY CONTROL DATA FROM 15/01/94 TO 21/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## CHLORIDE

### IDENTIFICATION:

|                      |   |                    |             |
|----------------------|---|--------------------|-------------|
| Laboratory Unit:     | Colourimetry  | Method Introduced: | 01/05/75    |
| Method Reference No: | E3016A  | Units:             | mg/L as Cl  |
| LIMS Product Code:   | CL3016  | Supervisor:        | M. Rawlings |
| Sample Type/Matrix:  | Rivers(non-APIOS), Lakes Soil Extracts, Effluents, Domestic Water Supplies, Leachates, Sewages, Industrial Wastes |                    |             |

### SAMPLING:

|                    |         |
|--------------------|---------|
| Quantity Required: | 10 mL   |
| Container:         | Plastic |

### ANALYTICAL PROCEDURE:

Chloride ions are combined with mercuric thiocyanate releasing thiocyanate quantitatively. Thiocyanate then reacts with ferric ions to produce ferric thiocyanate (red), and the absorbance of the latter is measured colourimetrically.

Approximate absorbance: 0.5 at the full scale level.

### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 1.5 cm light path at 480nm.

Data capture, reduction, and processing via a multistage microcomputer system.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

BL plus 10 standards

### CONTROLS:

|              |  |
|--------------|--|
| Calibration: | LTBL plus 3 standards, e.g. QCA                |
| Drift:       | BL every 10 samples; standard every 20 samples |

# CHLORIDE

QUALITY CONTROL DATA FROM 06/01/94 TO 23/12/94

Laboratory Unit: Colourimetry

Full Scale: to 100 mg/L as Cl

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 102 | 75.0                   | 75.2               | 0.2       | 0.2396                 |
| B:   | 102 | 25.0                   | 25.2               | 0.2       | 0.1225                 |
| C:   | 102 | 5.00                   | 5.04               | 0.04      | 0.2941                 |
| A+B: | 102 | 100.0                  | 100.3              | 0.3       | 0.2416                 |
| A-B: | 102 | 50.0                   | 50.2               | 0.2       | 0.0383                 |
| B+C: | 102 | 30.0                   | 30.2               | 0.2       | 0.1388                 |
| B-C: | 102 | 20.0                   | 20.1               | 0.1       | 0.1170                 |

s.d.(AB) S(between runs): 0.22

Sw(within run): 0.22

S/Sw: 1.0

s.d.(BC) S(between runs): 0.09

Sw(within run): 0.07

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |       |     |     |
|------|---|-------|-----|-----|
| 98.7 | - | 101.3 | for | A+B |
| 49.0 | - | 51.0  | for | A-B |
| 29.3 | - | 30.7  | for | B+C |
| 19.5 | - | 20.5  | for | B-C |

## DUPLICATES:

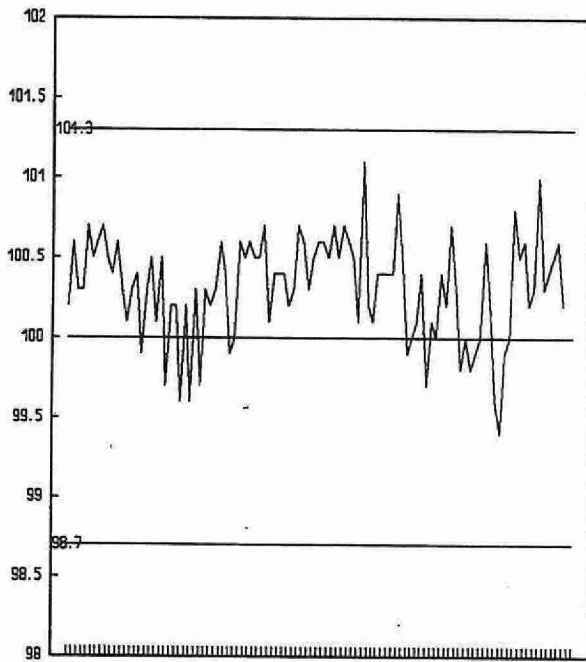
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 58           | 0.00 - 10.0               | 0.0901                 | 4.3                         |
| 64           | 10.1 - 20.0               | 0.1273                 | 0.9                         |
| 97           | 20.1 - 50.0               | 0.1790                 | 0.8                         |
| 42           | 50.1 - 100                | 0.3558                 | 0.6                         |
| 261          | Overall                   | 0.1678                 |                             |

## OTHER CHECKS:

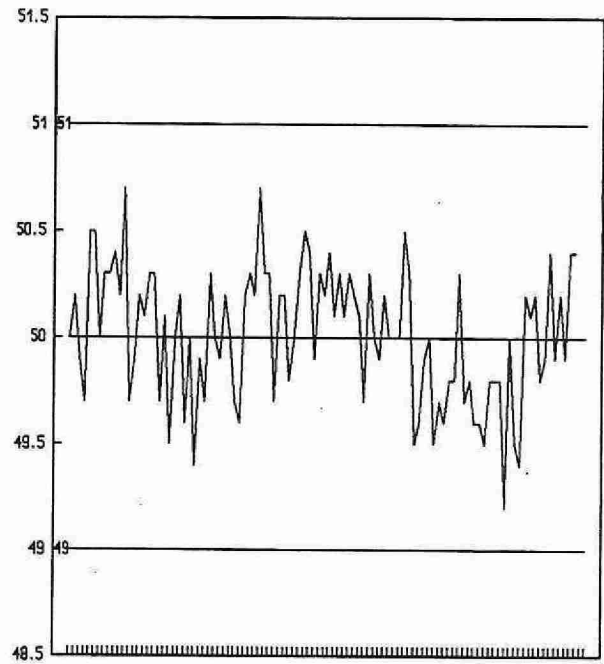
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 102 | -0.211 | 0.0922                 |

CHLORIDE (mg/L as Cl)

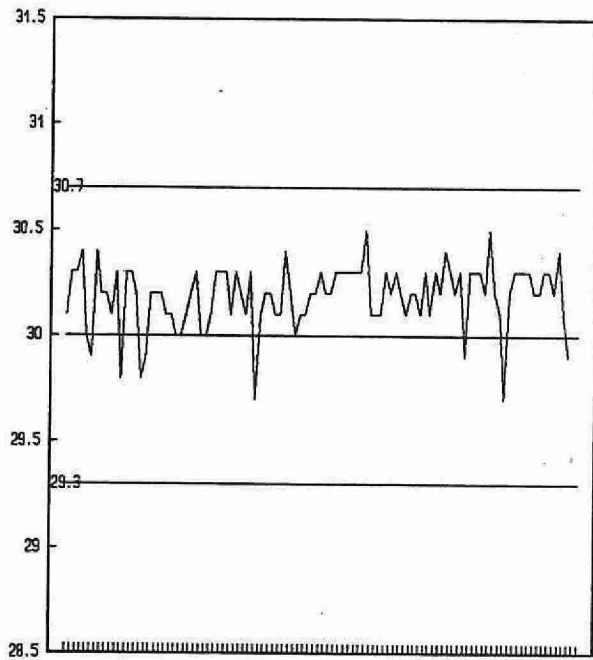
QUALITY CONTROL DATA FROM 06/01/94 TO 23/12/94



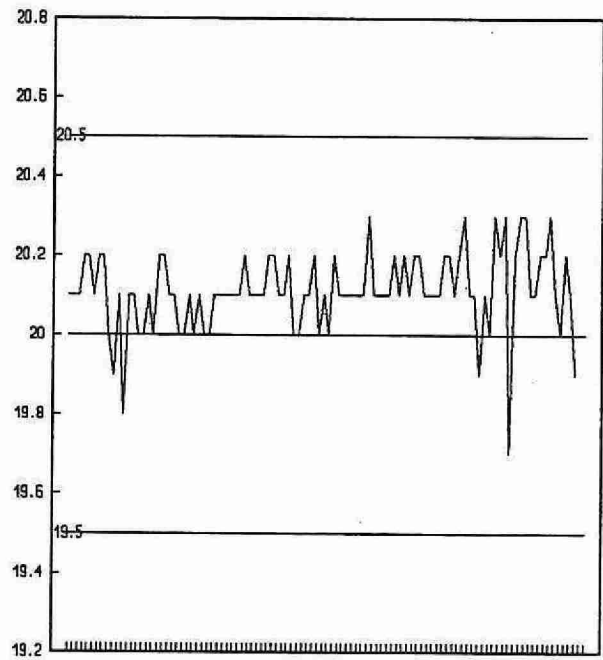
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## CHLORIDE

### IDENTIFICATION:

|                      |                                      |                   |            |
|----------------------|--------------------------------------|-------------------|------------|
| Laboratory Unit      | Dorset                               | Method Introduced | 01/04/78   |
| LIS Test Name Code   | CLIDUR                               | Units             | mg/L as Cl |
| Work Station Code    | DOIC                                 | Unit Code         | 064960     |
| Method Code          | 005AI0                               | Supervisor        | J. McBride |
| Method Reference No. | E3147A                               |                   |            |
| Sample Type/Matrix   | Precipitation, Throughfall, Stemflow |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 15 mL            |
| Container         | Glass or Plastic |

### ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with  $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$  to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample peak heights to a series of standards.

Nitrogen-nitrate and sulphate are determined simultaneously.

### INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.01 | Current T value: 0.05 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |                                  |
|-------------|----------------------------------|
| Calibration | LTBL plus 2 standards, e.g., QCA |
| Drift       | 1 standard every 10 samples.     |

# CHLORIDE

QUALITY CONTROL DATA FROM 07/01/94 TO 19/12/94

Laboratory Unit: Dorset

Full Scale: to 2.0 mg/L as Cl

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 35 | 1.60                   | 1.592              | -0.008    | 0.0238                 |
| B:   | 35 | 0.40                   | 0.397              | -0.003    | 0.0099                 |
| A+B: | 35 | 2.00                   | 1.997              | -0.003    | 0.0241                 |
| A-B: | 35 | 1.20                   | 1.195              | -0.005    | 0.0250                 |

s.d.(AB)      S(between runs): 0.018      Sw(within run): 0.018      S/Sw: 1.03

The calibration is accepted if the calibration control values obtained lie within the ranges:

1.93 - 2.07 for A+B  
1.14 - 1.26 for A-B

## DUPLICATES:

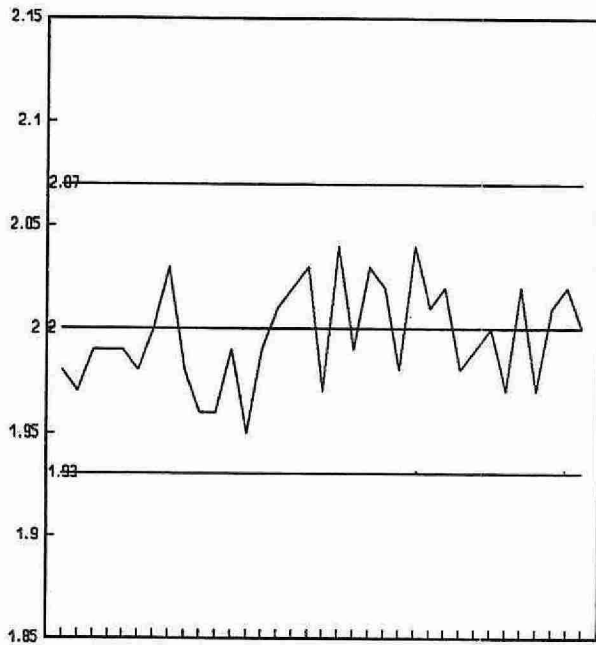
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 28           | 0.10 - 0.20               | 0.0091                 | 12.0                        |
| 50           | 0.21 - 0.40               | 0.0111                 | 3.9                         |
| 35           | 0.41 - 1.00               | 0.0144                 | 2.3                         |
| 10           | 1.01 - 2.00               | 0.0321                 | 2.0                         |
| 123          | Overall                   | 0.0124                 |                             |

## OTHER CHECKS:

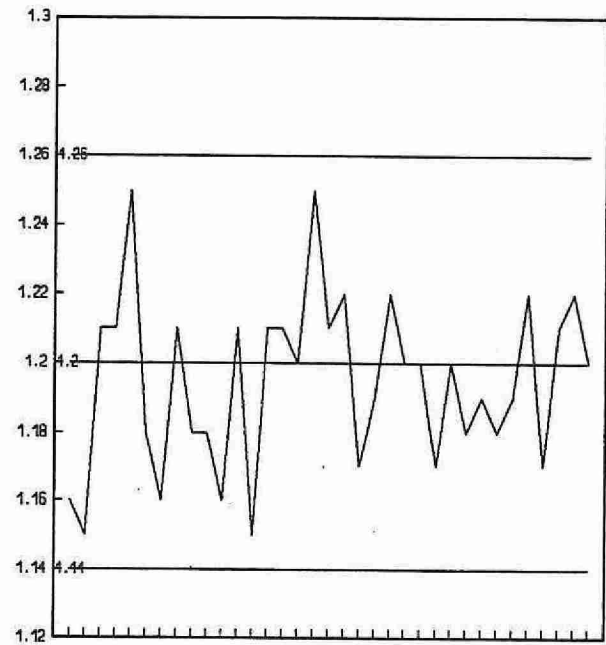
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 35 | -0.004 | 0.0088                 |

**CHLORIDE** (mg/L as Cl)

QUALITY CONTROL DATA FROM 07/01/94 TO 19/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## CHLORIDE

### IDENTIFICATION:

|                     |                                     |                   |                 |
|---------------------|-------------------------------------|-------------------|-----------------|
| Laboratory Unit     | Ion Chromatography                  | Method Introduced | 01/04/78        |
| Method Reference No | E3148A                              | Units             | µg/Filter as Cl |
| LIMS Product Code   | LOV3148, ANLOV3148                  | Supervisor        | F. Lo           |
| Sample Type/Matrix  | W40 filters from LoVol filter packs |                   |                 |

### SAMPLING:

|                   |                          |
|-------------------|--------------------------|
| Quantity Required | 1 filter                 |
| Container         | 50 mL polypropylene tube |

### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with  $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$  to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample peak heights to a series of standards. Results are converted to µg/filter as Cl. Nitrogen-nitrate and sulphate are determined simultaneously.

### INSTRUMENTATION:

Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

### REPORTING:

|                                |                            |                            |
|--------------------------------|----------------------------|----------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 mg/L | Current T value: 0.10 mg/L |
|--------------------------------|----------------------------|----------------------------|

### CALIBRATION:

BL plus 9 standards

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift       | 1 standard every 10 samples     |

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received. To convert unit from mg/L to µg/Filter, the concentration of Cl in mg/L is multiplied by 50 for the W40 filters.



# CHLORIDE

QUALITY CONTROL DATA FROM 19/01/94 TO 06/12/94

Laboratory Unit: Ion Chromatography

Full Scale: to 2.0 mg/L as Cl

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 40 | 1.60                   | 1.601              | 0.001     | 0.0093                 |
| B:   | 40 | 0.40                   | 0.4003             | 0.0003    | 0.0051                 |
| A+B: | 40 | 2.00                   | 2.001              | 0.001     | 0.0104                 |
| A-B: | 40 | 1.20                   | 1.201              | 0.001     | 0.0108                 |

s.d.(AB) S(between runs): 0.0075 Sw(within run): 0.0076 S/Sw: 0.99

The calibration is accepted if the calibration control values obtained lie within the ranges:

1.93 - 2.07 for A+B  
1.15 - 1.25 for A-B

## DUPLICATES:

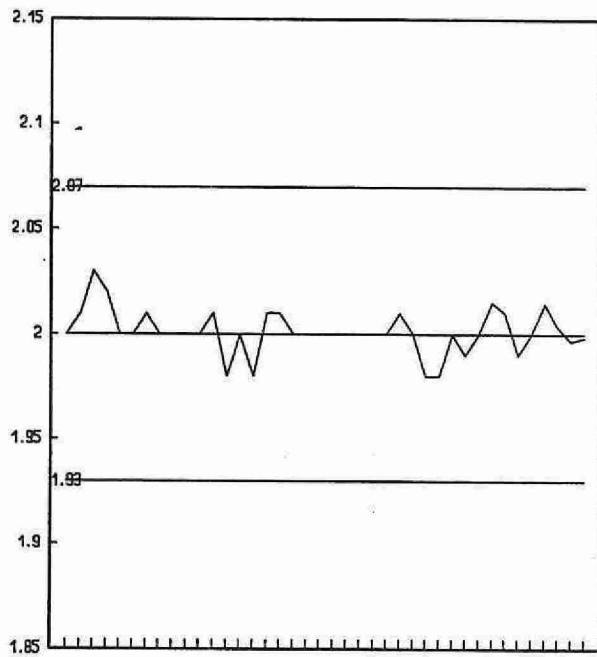
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 20           | 0.00 - 0.40               | 0.0030                 | 1.8                         |
| 14           | 0.41 - 1.00               | 0.0022                 | 0.5                         |
| 2            | 1.01 - 2.00               | 0.0064                 | 0.4                         |
| 36           | Overall                   | 0.0031                 |                             |

## OTHER CHECKS:

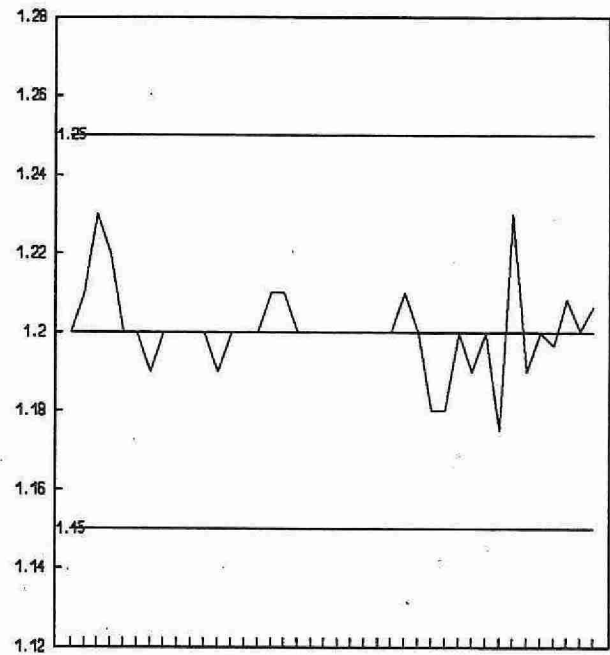
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 40 | 0.000 | 0.0000                 |

CHLORIDE (mg/L as Cl)

QUALITY CONTROL DATA FROM 19/01/94 TO 06/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## CHLORIDE

### IDENTIFICATION:

|                     |                                      |                   |            |
|---------------------|--------------------------------------|-------------------|------------|
| Laboratory Unit     | Ion Chromatography                   | Method Introduced | 01/04/78   |
| Method Reference No | E3372A                               | Units             | mg/L as Cl |
| LIMS Product Code   | ANION3372                            | Supervisor        | F. Lo      |
| Sample Type/Matrix  | Precipitation, Throughfall, Stemflow |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 15 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with  $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$  to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample peak heights to a series of standards.

Nitrogen-nitrate and sulphate are determined simultaneously.

### INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.01 | Current T value: 0.05 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift       | 1 standard every 10 samples     |

### NOTES:

Same analytical method as E3147A operating in Dorset Lab. New method number introduced for Toronto Lab in 1993 is E3372A.

# CHLORIDE

QUALITY CONTROL DATA FROM 11/01/94 TO 16/12/94

Laboratory Unit: Ion Chromatography

Full Scale: to 1.0 mg/L as Cl

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 34 | 0.80                   | 0.809              | 0.009     | 0.0101                 |
| B:   | 34 | 0.20                   | 0.201              | 0.001     | 0.0099                 |
| A+B: | 34 | 1.00                   | 1.009              | 0.009     | 0.0147                 |
| A-B: | 34 | 0.60                   | 0.608              | 0.008     | 0.0136                 |

s.d.(AB)      S(between runs): 0.0100      Sw(within run): 0.0096      S/Sw: 1.04

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.96 - 1.04 for A+B  
0.57 - 0.63 for A-B

## DUPLICATES:

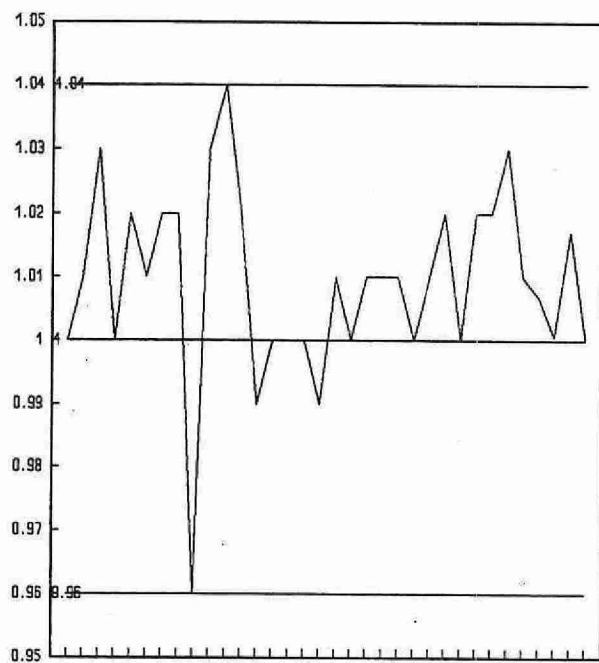
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 28           | 0.10 - 0.20               | 0.0058                 | 6.3                         |
| 4            | 0.21 - 0.50               | 0.0087                 | 3.1                         |
| 1            | 0.51 - 1.00               | N.A.                   | N.A.                        |
| 33           | Overall                   | 0.0063                 |                             |

## OTHER CHECKS:

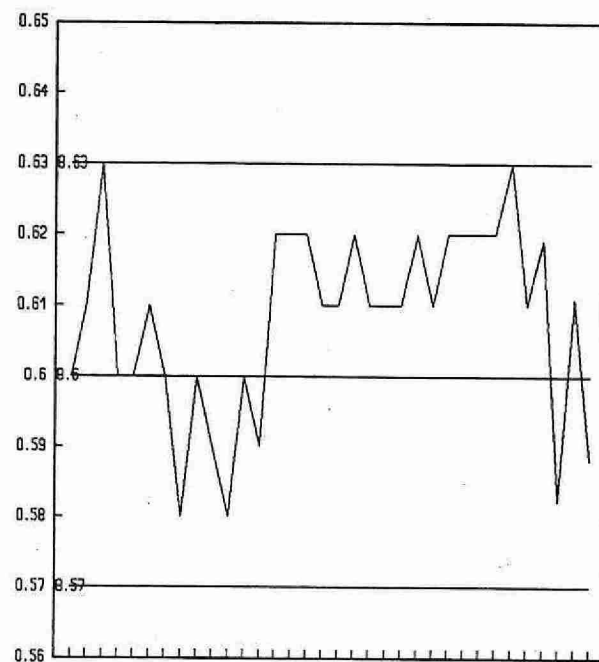
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 34 | 0.0081 | 0.0269                 |

CHLORIDE (mg/L as Cl)

QUALITY CONTROL DATA FROM 11/01/94 TO 16/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## CHLORINE, TOTAL RESIDUAL

### IDENTIFICATION:

|                      |   |                    |                         |
|----------------------|---|--------------------|-------------------------|
| Laboratory Unit:     | MISA  | Method Introduced: | 08/03/93                |
| Method Reference No: | E3309A  | Units:             | µg/L as Cl <sub>2</sub> |
| LIMS Product Code:   | RCL3309   | Supervisor:        | J. McBride              |
| Sample Type/Matrix:  | Industrial Waste , Sewage, Surface and Treated Drinking Water |                    |                         |

### SAMPLING:

|                    |                               |
|--------------------|-------------------------------|
| Quantity Required: | 1 L                           |
| Container:         | Narrow neck low actinic glass |

### ANALYTICAL PROCEDURE:

Samples are analyzed by amperometric titration. The sample pH is adjusted to between 3.5 and 4.5 with acetate buffer and excess KI is added.

### INSTRUMENTATION:

Autoburette

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |
|--------------------------------|--------------------|---------------------|

### CALIBRATION:

None

### CONTROLS:

|                   |                                  |
|-------------------|----------------------------------|
| Performance Check | QCBL plus 2 standards, e.g., QCA |
|-------------------|----------------------------------|

### NOTES:

Results recorded for duplicates are based upon final concentrations. The results from various sample aliquots are indicated in each of the concentration spans.

# CHLORINE, TOTAL RESIDUAL

QUALITY CONTROL DATA FROM 13/04/94 TO 22/12/94

Laboratory Unit: Colourimetry

Full Scale: to 50.0 µg/L as Cl<sub>2</sub>

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 22 | 40.0                      | 40.5                  | 0.5       | 1.4794                    |
| B:   | 22 | 10.0                      | 11.01                 | 1.01      | 1.3436                    |
| A+B: | 22 | 50.0                      | 51.5                  | 0.5       | 2.6483                    |
| A-B: | 22 | 30.0                      | 29.4                  | -0.4      | 0.9872                    |

s.d.(AB)

S(between runs):1.4

Sw(within run): 0.7

S/Sw: 2.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

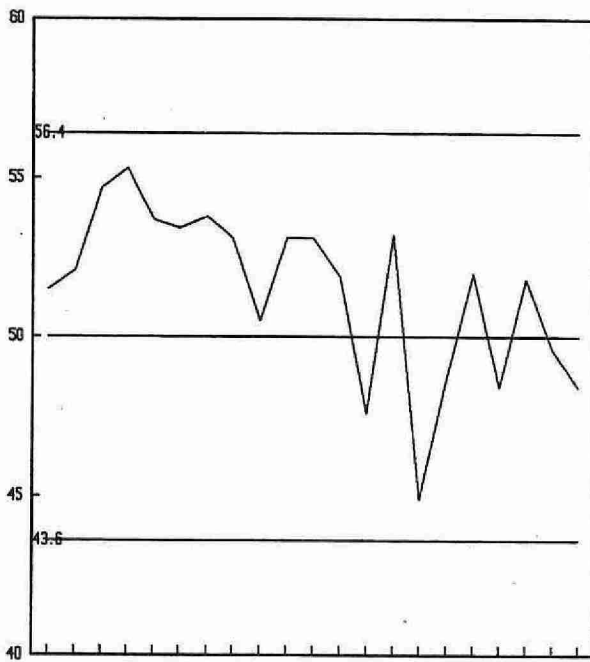
43.6 - 56.4 for A+B  
25.2 - 34.8 for A-B

## DUPLICATES:

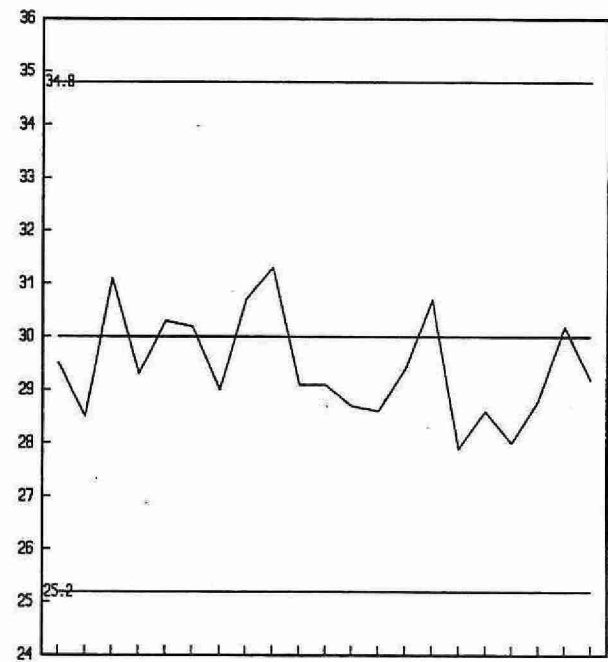
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 2               | 0 - 10                       | N.A.                      | N.A.                           |
| 2               | 11 - 25                      | N.A.                      | N.A.                           |
| 4               | 26 - 50                      | 0.2748                    | 0.73                           |
| 4               | 51 - 100                     | 3.4542                    | 4.7                            |
| 7               | 101 - 700                    | 21.486                    | 4.6                            |
| 19              | Overall                      | 9.578                     |                                |

# CHLORINE TOTAL RESIDUAL ( $\mu\text{g/L}$ as $\text{Cl}_2$ )

QUALITY CONTROL DATA FROM 13/04/94 TO 22/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT



## CHLOROPHYLL

### IDENTIFICATION:

|                      |                          |                    |             |
|----------------------|--------------------------|--------------------|-------------|
| Laboratory Unit:     | Colourimetry             | Method Introduced: | 01/04/75    |
| Method Reference No: | E3169A                   | Units:             | µg/L        |
| LIMS Product Code:   | CHL3169                  | Supervisor:        | M. Rawlings |
| Sample Type/Matrix:  | Rivers, Lakes, Effluents |                    |             |

### SAMPLING:

|                   |   |
|-------------------|---|
| Quantity Required | 1000 mL for clear samples; 500 mL if visibly green  |
| Container         | Glass or plastic  |
| Other             | In the field a sample is filtered through a nylon filter. The filter is folded and then placed between two membrane filter-support pads, and the package is enclosed in a plastic dish labelled with the sample number and sample volume filtered, the dish is kept in the dark or wrapped in aluminum foil, and shipped immediately, or kept frozen. |

### ANALYTICAL PROCEDURE:

Using a Commodore PET microcomputer-controlled, automated spectrophotometer, two scans are developed with absorbance measurements at 630, 645, and 663 nm for the first scans; the minimum absorbance value between 710 and 750 nm (readings at 5 nm intervals) is utilized as a turbidity correction. Chlorophyll "a" and "b" are calculated from this scan. After automated acidification, the second scan is obtained from the wavelength 665 nm for correcting chlorophyll "a" measurement. SCOR-UNESCO equations are used for all chlorophyll calculations.

### INSTRUMENTATION:

- Automated modular continuous flow scanning spectrophotometer system
- Microcomputer system for control of sampling, timing and data processing (i.e. data capture, calculations and transfer of results to LIS)

### REPORTING:

|                          |                     |                      |                      |
|--------------------------|---------------------|----------------------|----------------------|
| Chlorophyll a; corrected | Maximum Significant | Current W value: 1.0 | Current T value: 5.0 |
| Chlorophyll a; total     | Figures: 3          | Current W value: 0.2 | Current T value: 1.0 |
| Chlorophyll b; total     |                     | Current W value: 0.1 | Current T value: 0.5 |

### CONTROLS:

|             |                                  |
|-------------|----------------------------------|
| Calibration | LTBL plus 2 "standards", e.g.QCA |
| Drift       | "standard", BL every 20 samples  |

### NOTES:

"Standards" are prepared from chlorophyll "a" and "b", but the materials are neither analytical grade nor are their solutions stable. Thus calibration controls are based on measured averages.

# CHLOROPHYLL "a"

QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94

Laboratory Unit: Colourimetry

Full Scale: to 50 µg/L

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 135 | 3.0                    | 2.997              | -0.003    | 0.0953                 |
| B:   | 135 | 1.0                    | 1.007              | 0.007     | 0.0633                 |
| A+B: | 135 | 4.0                    | 4.004              | 0.004     | 0.1370                 |
| A-B: | 135 | 2.0                    | 1.990              | -0.010    | 0.0859                 |

s.d.(AB)

S(between runs): 0.08

Sw(within run): 0.06

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

3.60 - 4.40 for A+B  
1.80 - 2.20 for A-B

## DUPLICATES:

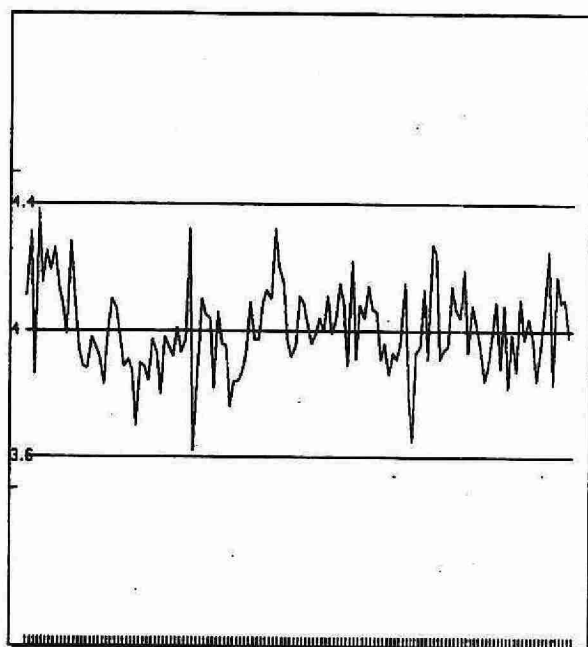
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 126          | 0.0 - 5.0                 | 0.2463                 | 40.4                        |
| 28           | 5.1 - 10.0                | 0.6041                 | 19.3                        |
| 24           | 10.1 - 25.0               | 2.4940                 | 17.8                        |
| 9            | 25.1 - 50.0               | 2.8158                 | 11.5                        |
| 187          | Overall                   | 0.4492                 |                             |

## OTHER CHECKS:

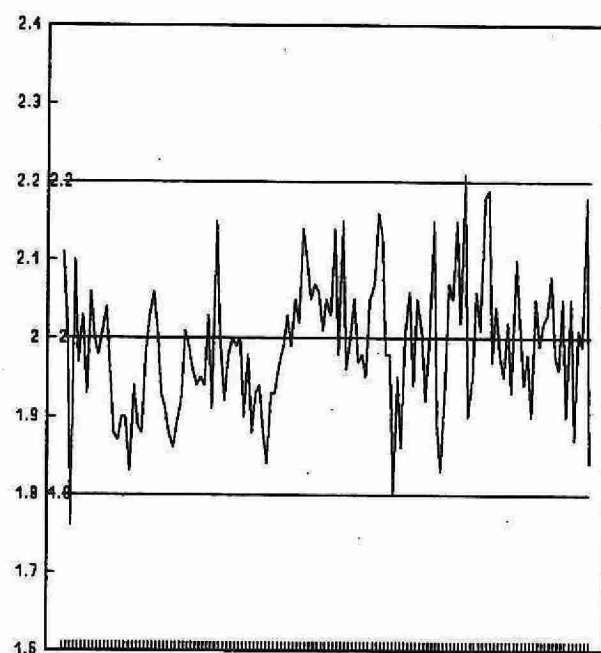
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 135 | 0.0584 | 0.0719                 |

# CHLOROPHYLL, "a" ( $\mu\text{g/L}$ )

QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

# CHLOROPHYLL "a", ACIDIFIED

QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94

Laboratory Unit: Colourimetry

Full Scale: to 10.0 µg/L

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 135 | 2.4                    | 2.505              | 0.105     | 0.2468                 |
| B:   | 135 | 0.8                    | 0.797              | -0.003    | 0.1382                 |
| A+B: | 135 | 3.2                    | 3.302              | 0.102     | 0.3391                 |
| A-B: | 135 | 1.6                    | 1.708              | 0.108     | 0.2274                 |

s.d.(AB) S(between runs): 0.20

Sw(within run): 0.16

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.4 - 4.0 for A+B  
1.1 - 2.1 for A-B

## DUPLICATES:

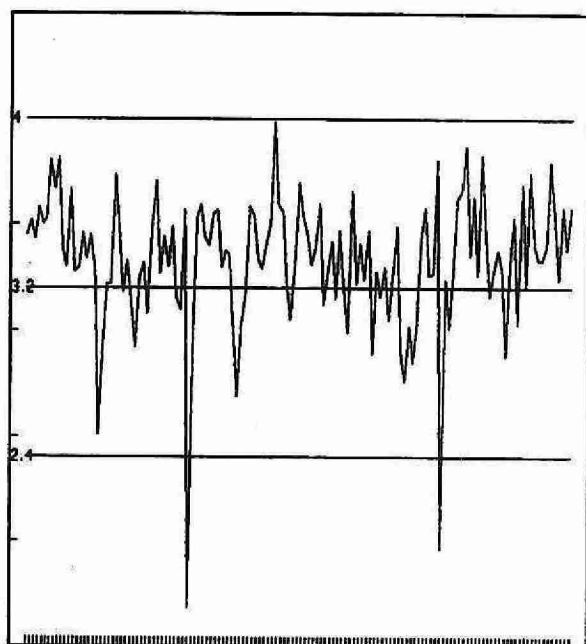
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 53           | -6.3 - 1.0                | 0.7104                 | 464.5                       |
| 19           | 1.1 - 2.0                 | 0.7384                 | 88.8                        |
| 16           | 2.1 - 5.0                 | 1.0002                 | 33.2                        |
| 7            | 5.1 - 10.0                | 2.3443                 | 30.9                        |
| 96           | Overall                   | 0.8345                 |                             |

## OTHER CHECKS:

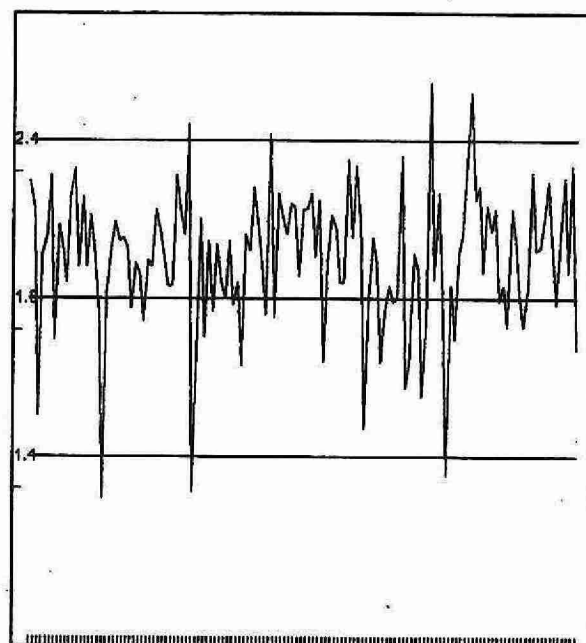
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 135 | -0.052 | 0.1450                 |

**CHLOROPHYLL "a", ACIDIFIED ( $\mu\text{g/L}$ )**

QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

# CHLOROPHYLL "b"

QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94

Laboratory Unit: Colourimetry

Full Scale: to 10.0 µg/L

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 135 | 3.0                    | 2.998              | -0.002    | 0.1176                 |
| B:   | 135 | 1.0                    | 1.005              | 0.005     | 0.1127                 |
| A+B: | 135 | 4.0                    | 4.003              | 0.003     | 0.2051                 |
| A-B: | 135 | 2.0                    | 1.993              | -0.007    | 0.1049                 |

s.d.(AB)

S(between runs): 0.12

Sw(within run): 0.07

S/Sw: 1.6

The calibration is accepted if the calibration control values obtained lie within the ranges:

3.60 - 4.40 for A+B  
1.80 - 2.20 for A-B

## DUPLICATES:

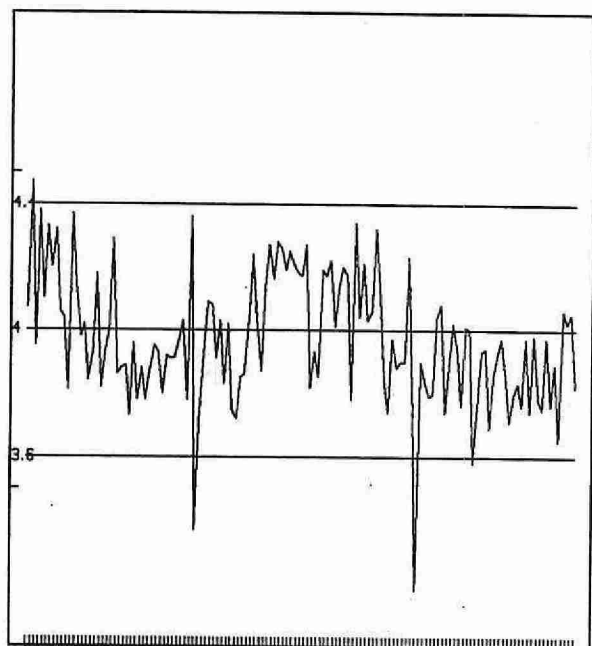
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 106          | 0.0 - 1.0                 | 0.2047                 | 48.1                        |
| 37           | 1.1 - 2.0                 | 0.2702                 | 35.8                        |
| 18           | 2.1 - 5.0                 | 0.4232                 | 14.5                        |
| 2            | 5.1 - 10.0                | N.A.                   | N.A.                        |
| 163          | Overall                   | 0.2380                 |                             |

## OTHER CHECKS:

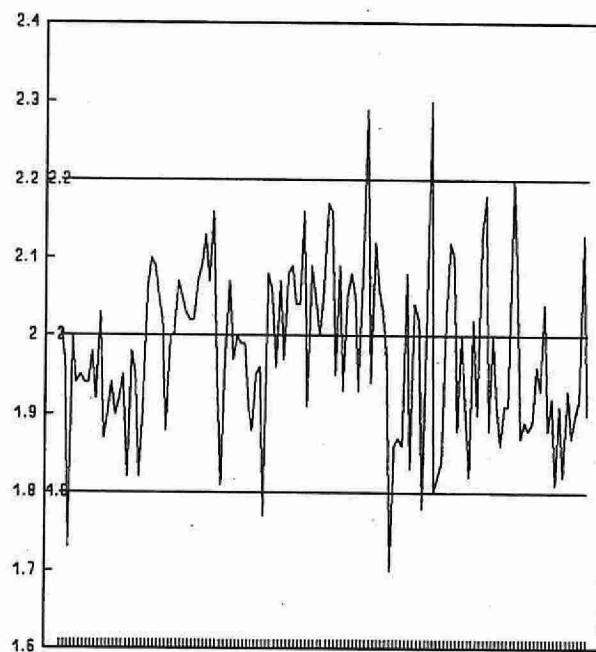
|                 | n   | Mean  | Standard Deviation (1) |
|-----------------|-----|-------|------------------------|
| Long Term Blank | 135 | 0.074 | 0.1275                 |

CHLOROPHYLL, "b" ( $\mu\text{g/L}$ )

QUALITY CONTROL DATA FROM 11/01/94 TO 22/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## COLOUR, TRUE

### IDENTIFICATION:

|                      |                |                   |            |
|----------------------|----------------|-------------------|------------|
| Laboratory Unit      | Dorset         | Method Introduced | 15/10/80   |
| LIS Test Name Code   | COLTR          | Units             | TCU        |
| Work Station Code    | DOCOL          | Unit Code         | 340000     |
| Method Code          | 102BC9         | Supervisor        | J. McBride |
| Method Reference No. | E3025A         |                   |            |
| Sample Type/Matrix:  | Streams, Lakes |                   |            |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: | 25 mL            |
| Container:         | Glass or plastic |

### ANALYTICAL PROCEDURE:

True colour is measured on a settled sample colourimetrically in a system calibrated with acidified chloroplatinate standards. Colour is measured using a 400-450 nm broadband blue filter.  
Approximate absorbance: 0.20 at the full scale level.

### INSTRUMENTATION:

One colourimeter with broadband blue filter (400-450 nm)  
One autosampler and chart-recorder  
One Gilson pump

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

6 acidified chloroplatinate standards, 10, 20, 40, 60, 80, 100 TCU

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 standards, e.g. QCA, QCB, QCC |
|-------------|---|

### NOTES:

Slope factor is changed whenever light source in a colourimeter or cell is replaced. This is accomplished by analyzing 7 standards.



# COLOUR, TRUE

QUALITY CONTROL DATA FROM 10/01/94 TO 20/12/94

Laboratory Unit: Dorset

Analytical Range: to 100 TCU

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 39 | 75                     | 75.61              | 0.61      | 0.5362                 |
| B:   | 39 | 25                     | 25.59              | 0.59      | 0.4553                 |
| C:   | 39 | 5                      | 5.51               | 0.51      | 0.3768                 |
| A+B: | 39 | 50                     | 50.02              | 0.02      | 0.4283                 |
| A-B: | 39 | 100                    | 100.09             | 0.09      | 0.5748                 |
| B+C: | 39 | 30                     | 29.99              | -0.01     | 0.3592                 |
| B-C: | 39 | 20                     | 20.08              | 0.08      | 0.4336                 |

s.d.(AB) S(between runs): 0.50 Sw(within run): 0.30 S/Sw: 1.6

s.d.(BC) S(between runs): 0.42 Sw(within run): 0.31 S/Sw: 1.4

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |       |     |     |
|------|---|-------|-----|-----|
| 96.7 | - | 103.3 | for | A+B |
| 47.5 | - | 52.5  | for | A-B |
| 27.7 | - | 32.3  | for | B+C |
| 18.2 | - | 21.8  | for | B-C |

## DUPLICATES:

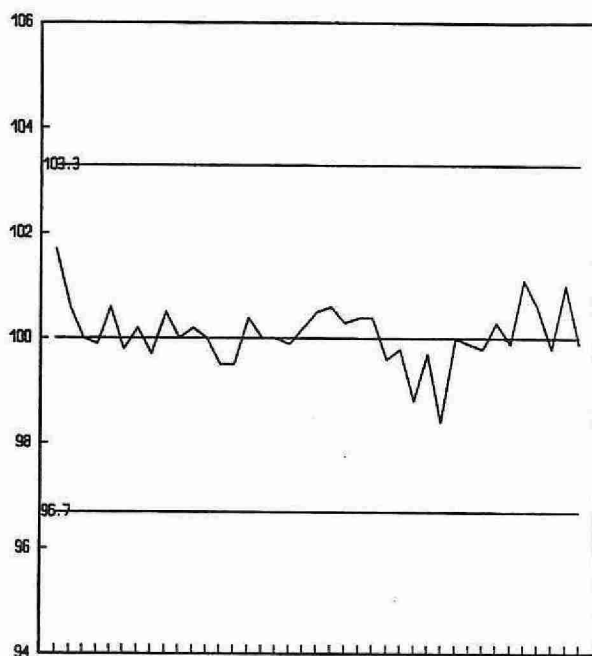
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 11           | 0.0 - 10.0                | 0.2907                 | 3.9                         |
| 26           | 10.1 - 20.0               | 0.4299                 | 2.7                         |
| 55           | 20.1 - 50.0               | 0.5669                 | 1.5                         |
| 22           | 50.1 - 100.0              | 1.1237                 | 1.6                         |
| 114          | Overall                   | 0.5824                 |                             |

## OTHER CHECKS:

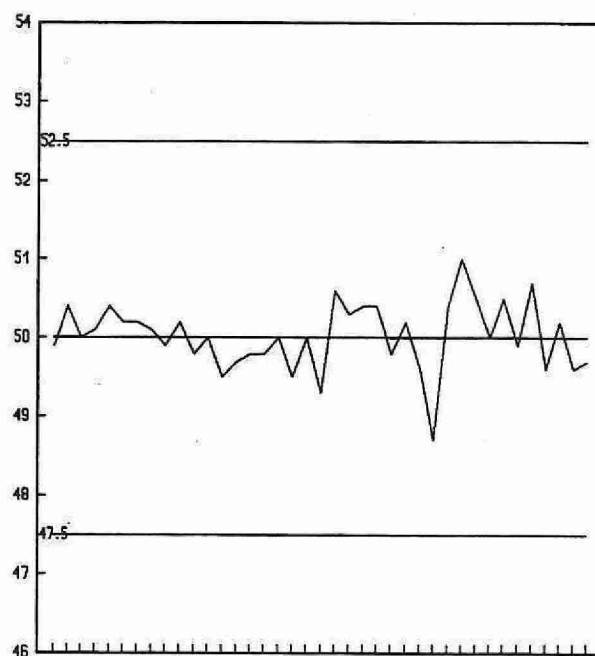
|                 | n  | Data Mean | Standard (1) Deviation |
|-----------------|----|-----------|------------------------|
| Long Term Blank | 39 | 0.556     | 0.3362                 |

# COLOUR, TRUE (TCU)

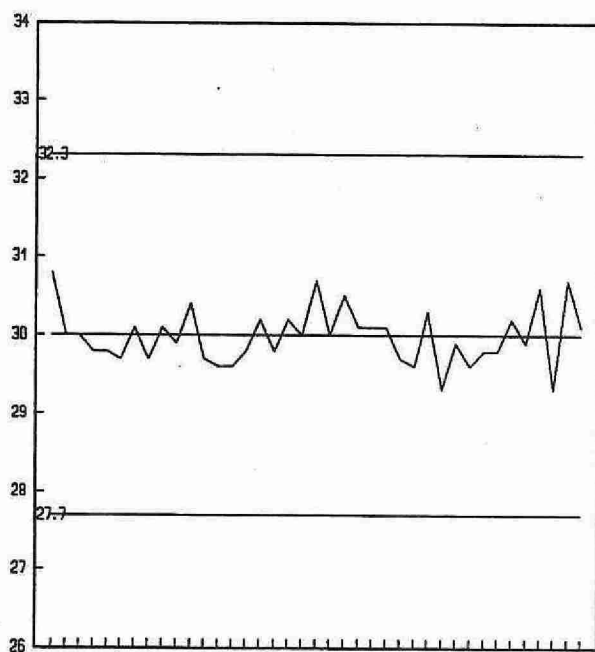
QUALITY CONTROL DATA FROM 10/01/94 TO 20/12/94



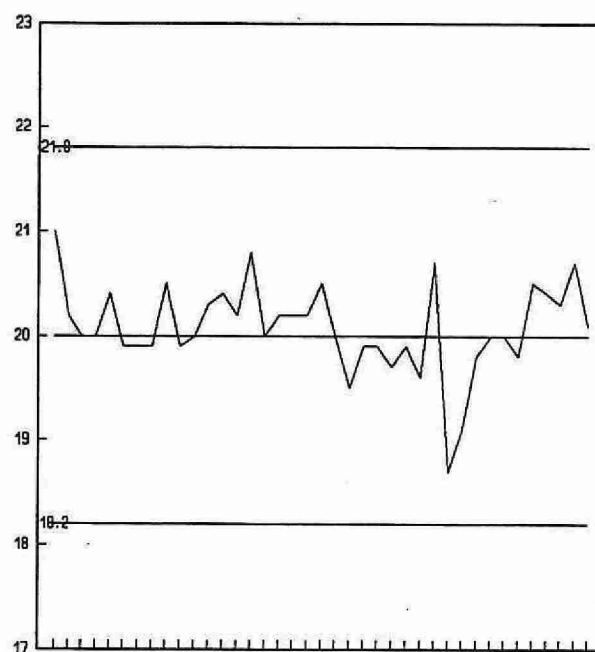
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## COLOUR, TRUE

### IDENTIFICATION:

|                     |  |                   |             |
|---------------------|--|-------------------|-------------|
| Laboratory Unit     | Colourimetry   | Method Introduced | 13/03/84    |
| Method Reference No | E3219A   | Units             | TCU         |
| LIMS Product Code   | COL3219  | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Domestic Waters, Effluents, Surface Waters, Industrial Wastes, Leachates |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

True colour is measured colourimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference stream.

Approximate absorbance: 0.3 at the full scale level.

### INSTRUMENTATION:

Basic automated modular continuous flow system. Colour measurement is through a 3.0 cm. light path using a broadband filter (400-450 nm). Turbidity measurement is through a 5.0 cm. light path using a different broadband filter (660-740 nm). Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

BL plus 6 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 2 standards, e.g. QCA                |
| Drift       | BL every 10 samples; standard every 20 samples |

# COLOUR, TRUE

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94

Laboratory Unit: Colourimetry

Full Scale: to 100 TCU

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 44 | 70.0                   | 70.7               | 0.7       | 0.4409                 |
| B:   | 44 | 25.0                   | 24.8               | -0.2      | 0.3684                 |
| C:   | 44 | 7.50                   | 7.505              | 0.005     | 0.3299                 |
| A+B: | 44 | 95.0                   | 95.5               | 0.5       | 0.6711                 |
| A-B: | 44 | 45.0                   | 45.9               | 0.9       | 0.4580                 |
| B+C: | 44 | 32.5                   | 32.3               | -0.2      | 0.6112                 |
| B-C: | 44 | 17.5                   | 17.3               | -0.2      | 0.3399                 |

s.d.(AB) S(between runs): 0.41

Sw(within run): 0.32

S/Sw: 1.3

s.d.(BC) S(between runs): 0.35

Sw(within run): 0.24

S/Sw: 1.5

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 92.2 | - | 97.8 | for | A+B |
| 42.9 | - | 47.1 | for | A-B |
| 30.6 | - | 34.3 | for | B+C |
| 16.1 | - | 18.9 | for | B-C |

## DUPLICATES:

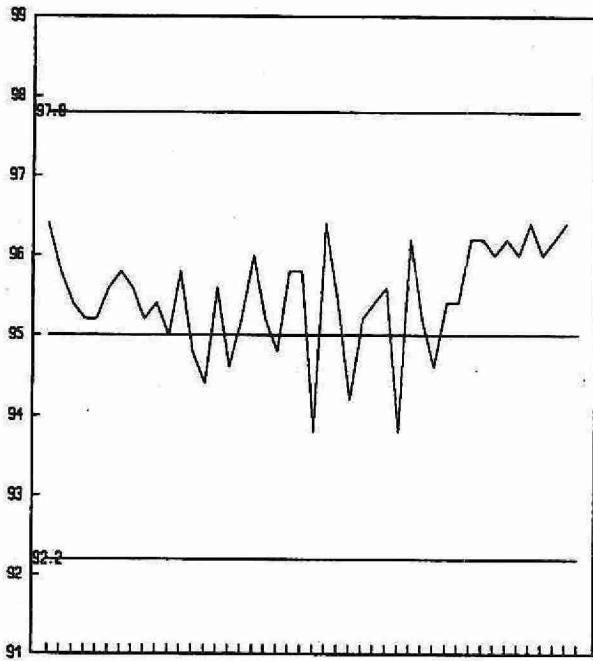
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 104          | 0.00 - 10.0               | 0.2577                 | 16.5                        |
| 18           | 10.1 - 20.0               | 0.2878                 | 1.7                         |
| 8            | 20.1 - 50.0               | 0.4975                 | 1.6                         |
| 1            | 50.1 - 100                | N.A.                   | N.A.                        |
| 131          | Overall                   | 0.2846                 |                             |

## OTHER CHECKS:

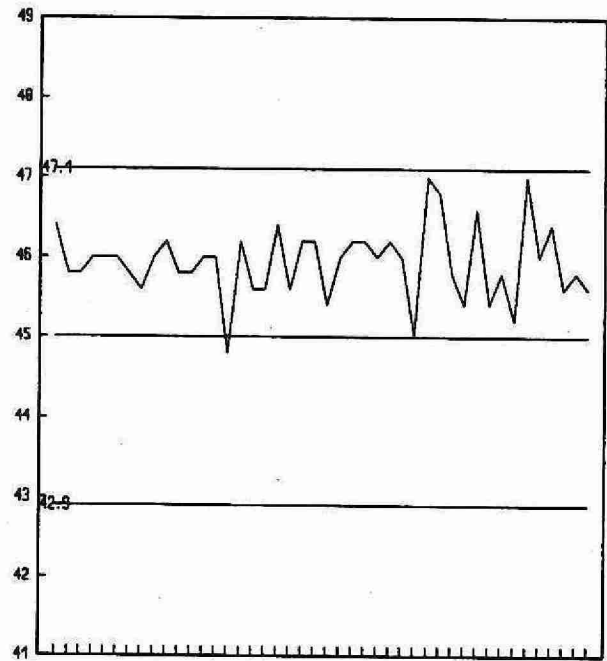
|                 | n  | Mean    | Standard Deviation (1) |
|-----------------|----|---------|------------------------|
| Long Term Blank | 44 | -0.0046 | 0.5921                 |

# COLOUR, TRUE (TCU)

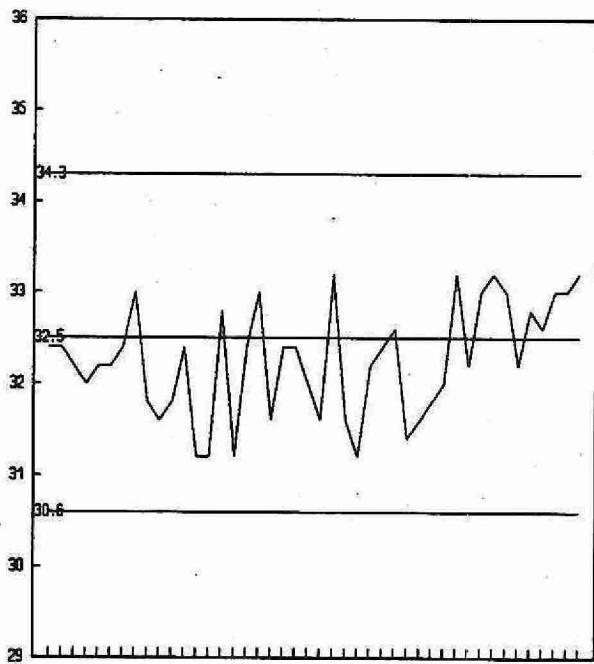
QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94



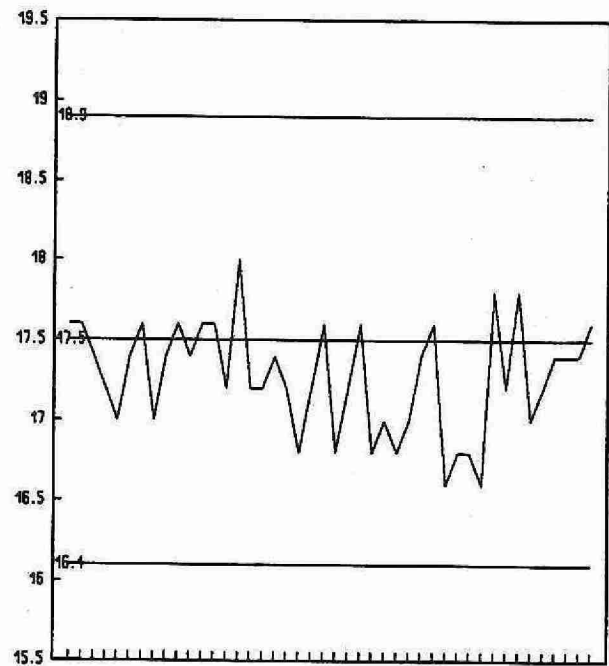
QUALITY CONTROL SAMPLE A+B



QUALITY CONTROL SAMPLE A-B



QUALITY CONTROL SAMPLE B+C



QUALITY CONTROL SAMPLE B-C

CONTROL LIMIT

## CONDUCTIVITY

### IDENTIFICATION:

|                      |   |                   |                                 |
|----------------------|---|-------------------|---------------------------------|
| Laboratory Unit      | Dorset  | Method Introduced | 01/06/76                        |
| LIS Test Name Code   | COND25  | Units             | $\mu\text{S}/\text{cm}$ at 25°C |
| Work Station Code    | DOCOND  | Unit Code         | 350351                          |
| Method Code          | 002BI2  | Supervisor        | J. McBride                      |
| Method Reference No. | E3024B  |                   |                                 |
| Sample Type/Matrix   | Streams, Lakes, Precipitation, Soil Leachates |                   |                                 |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 75 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

The sample is introduced into a jacketed conductivity cell. The conductivity is calculated from the chart record.

### INSTRUMENTATION:

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator. One autosampler, Gilson pump and dual-range chart recorder.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

5 KCl standards, 10.2, 30.6, 50.8, 101.1, 151 S

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 4 standards, e.g. QCA |
|-------------|---------------------------------|

### NOTES:

The control standards are corrected for the LTB from which they are made.

# CONDUCTIVITY

QUALITY CONTROL DATA FROM 10/01/94 TO 22/12/94

Laboratory Unit: Dorset

Analytical Range: to 500  $\mu\text{S}/\text{cm}$

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 48 | 146.7                  | 147.04             | 0.34      | 0.6444                 |
| B:   | 48 | 51.8                   | 52.82              | 1.02      | 0.7793                 |
| C:   | 48 | 51.8                   | 52.70              | 0.90      | 0.6279                 |
| D:   | 48 | 14.9                   | 15.52              | 0.62      | 0.2641                 |
| A+B: | 48 | 198.5                  | 198.95             | 0.45      | 1.0143                 |
| A-B: | 48 | 94.9                   | 94.23              | -0.67     | 1.0766                 |
| C+D: | 48 | 66.7                   | 67.31              | 0.61      | 0.6385                 |
| C-D: | 48 | 36.9                   | 37.18              | 0.28      | 0.5908                 |

s.d.(AB) S(between runs): 0.72

Sw(within run): 0.76

S/Sw: 0.94

s.d.(CD) S(between runs): 0.48

Sw(within run): 0.42

S/Sw: 1.15

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 195  | - | 202  | for | A+B |
| 92.2 | - | 97.6 | for | A-B |
| 64.9 | - | 68.5 | for | C+D |
| 35.6 | - | 38.2 | for | C-D |

## DUPLICATES:

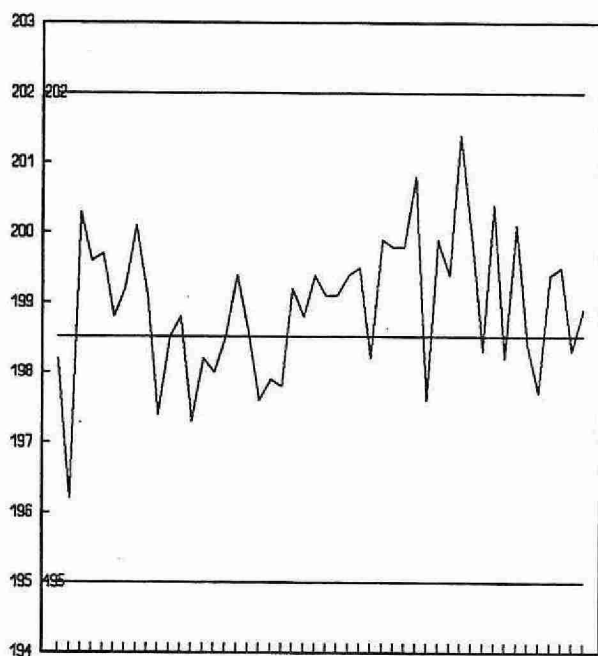
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 129          | 0.00 - 50.0               | 0.7289                 | 3.0                         |
| 10           | 50.1 - 100.0              | 0.4632                 | 0.7                         |
| 5            | 100.1 - 250.0             | 0.0000                 | 1.6                         |
| 2            | 250.1 - 500.0             | N.A.                   | N.A.                        |
| 146          | Overall                   | 0.7117                 |                             |

## OTHER CHECKS:

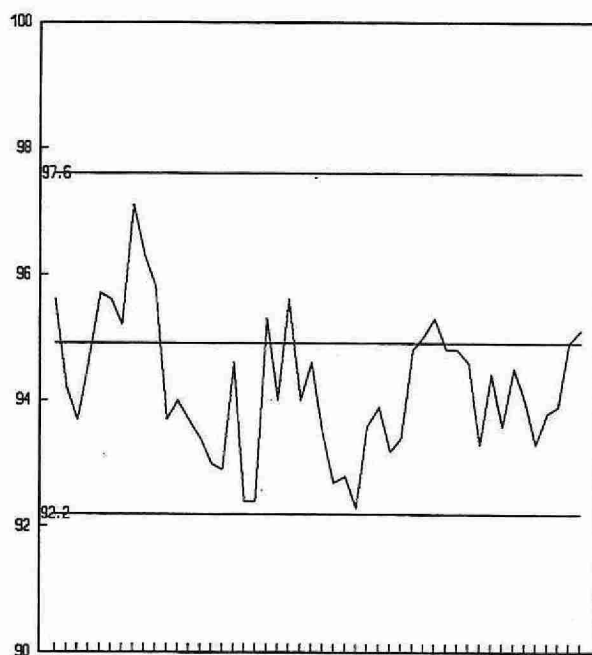
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 48 | 0.4542 | 0.3162                 |

# CONDUCTIVITY ( $\mu\text{S}/\text{cm}$ )

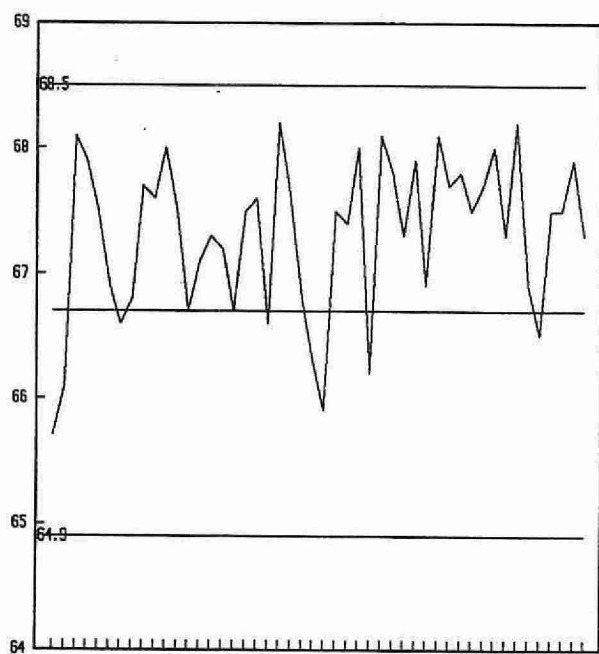
QUALITY CONTROL DATA FROM 10/01/94 TO 22/12/94



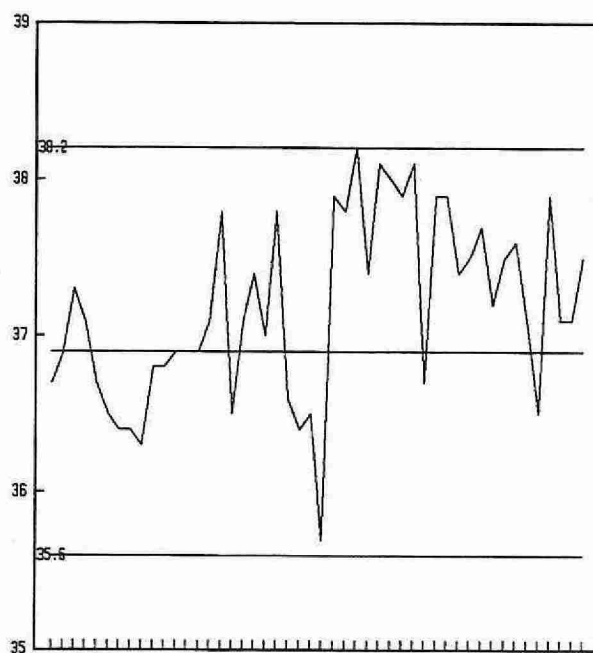
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT



## CONDUCTIVITY

### IDENTIFICATION:

|                      |                                      |                    |                                 |
|----------------------|--------------------------------------|--------------------|---------------------------------|
| Laboratory Unit:     | Ion Chromatography                   | Method Introduced: | 01/04/78                        |
| Method Reference No: | E3177A                               | Units:             | $\mu\text{S}/\text{cm}$ at 25°C |
| LIMS Product Code:   | COND3177                             | Supervisor:        | F. Lo                           |
| Sample Type/Matrix:  | Precipitation, Throughfall, Stemflow |                    |                                 |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: | 15 mL            |
| Container:         | Glass or plastic |

### ANALYTICAL PROCEDURE:

After equilibration at 25°C, The conductivity of the sample is measured.

### INSTRUMENTATION:

Automated modular continuous flow conductivity system comprised of sampler, water bath, conductivity meter with cell, chart recorder.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

1 standard

### CONTROLS:

|              |                                 |
|--------------|---------------------------------|
| Calibration: | LTBL plus 2 standards, e.g. QCA |
| Drift:       | 1 solution every 10 samples     |

### NOTES:

A calibration standard for the ion chromatographic system is used to monitor the drift for the conductivity system, but its theoretical conductivity is unknown.

# CONDUCTIVITY

QUALITY CONTROL DATA FROM 11/01/94 TO 26/09/94

Laboratory Unit: Ion Chromatography

Full Scale: to 100.0  $\mu\text{S}/\text{cm}$

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 27 | 44.5                   | 45.3               | 0.8       | 0.6097                 |
| B:   | 27 | 7.5                    | 8.0                | 0.5       | 0.3650                 |
| A+B: | 27 | 52.0                   | 53.3               | 1.3       | 0.6815                 |
| A-B: | 27 | 37.0                   | 37.2               | 0.2       | 0.7386                 |

s.d.(AB)    S(between runs): 0.50                      Sw(within run): 0.52                      S/Sw: 0.96

The calibration is accepted if the calibration control values obtained lie within the ranges:

47.76   -   56.24    for   A+B  
33.82   -   40.18    for   A-B

## DUPLICATES:

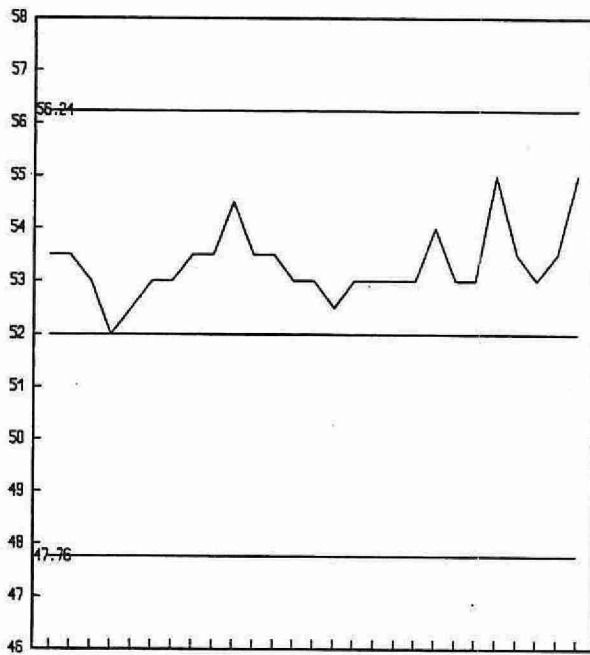
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 9               | 0.0 - 20.0                   | 0.2635                    | 2.1                            |
| 21              | 20.1 - 50.0                  | 0.4404                    | 1.5                            |
| 1               | 50.1 - 100.0                 | N.A.                      | N.A.                           |
| 31              | Overall                      | 0.3716                    |                                |

## OTHER CHECKS:

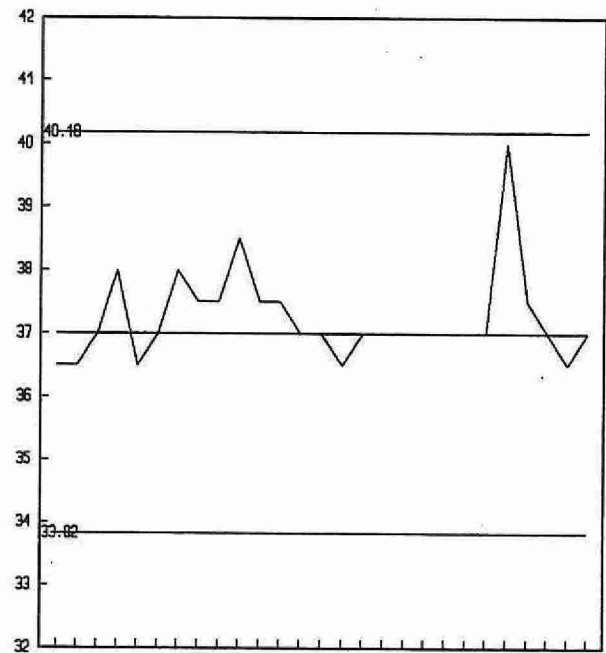
|                 | n  | Mean   | Standard<br>Deviation (1) |
|-----------------|----|--------|---------------------------|
| Long Term Blank | 27 | 0.7963 | 0.3180                    |

# CONDUCTIVITY ( $\mu\text{S}/\text{cm}$ )

QUALITY CONTROL DATA FROM 11/01/94 TO 26/09/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

\_\_\_\_\_ CONTROL LIMIT

## CONDUCTIVITY

### IDENTIFICATION:

|                      |   |                    |                                 |
|----------------------|---|--------------------|---------------------------------|
| Laboratory Unit:     | Titration                                     | Method Introduced: | 01/04/74                        |
| Method Reference No: | E3218A  | Units:             | $\mu\text{S}/\text{cm}$ at 25°C |
| LIMS Product Code:   | PHALCO3218,CONDPH3218                         | Supervisor:        | F. Lo                           |
| Sample Type/Matrix:  | Domestic Waters, Sewage, Industrial effluents |                    |                                 |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: | 25 mL            |
| Container:         | Glass or plastic |

### ANALYTICAL PROCEDURE:

After equilibration at 25°C, the conductivity of the sample is measured. pH, and total fixed endpoint alkalinity are determined simultaneously.

### INSTRUMENTATION:

Automated modular continual flow conductivity system comprising of a sampler, water bath, pump, conductivity meter with cell plus microcomputer control and data processing software.

### REPORTING:

|                                |                    |                    |
|--------------------------------|--------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 1 | Current T value: 5 |
|--------------------------------|--------------------|--------------------|

### CONTROLS:

|              |  |
|--------------|--|
| Calibration: | LTBL plus 3 standards, e.g. QCA                                    |
| Drift:       | In run standards throughout the run (tap water diluted to 50% V/V) |

# CONDUCTIVITY

QUALITY CONTROL DATA FROM 04/01/94 TO 21/12/94

Laboratory Unit: Titration

Analytical Range: to 2000  $\mu\text{S}/\text{cm}$

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 97 | 1413.0                    | 1408.3                | -4.7      | 5.0021                    |
| B:   | 97 | 717.8                     | 716.6                 | -1.2      | 1.9574                    |
| C:   | 97 | 717.8                     | 716.6                 | -1.2      | 1.9574                    |
| D:   | 97 | 147.0                     | 147.6                 | 0.6       | 0.7212                    |
| A+B: | 97 | 2130.8                    | 2124.8                | -6.0      | 6.4410                    |
| A-B: | 97 | 695.2                     | 691.7                 | -3.5      | 4.0274                    |
| C+D: | 97 | 864.8                     | 864.1                 | -0.7      | 2.4163                    |
| C-D: | 97 | 570.8                     | 569.0                 | -1.8      | 1.6925                    |

s.d.(AB)    S(between runs): 3.80                      Sw(within run): 2.85                      S/Sw: 1.3

s.d.(CD)    S(between runs): 1.48                      Sw(within run): 1.20                      S/Sw: 1.2

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

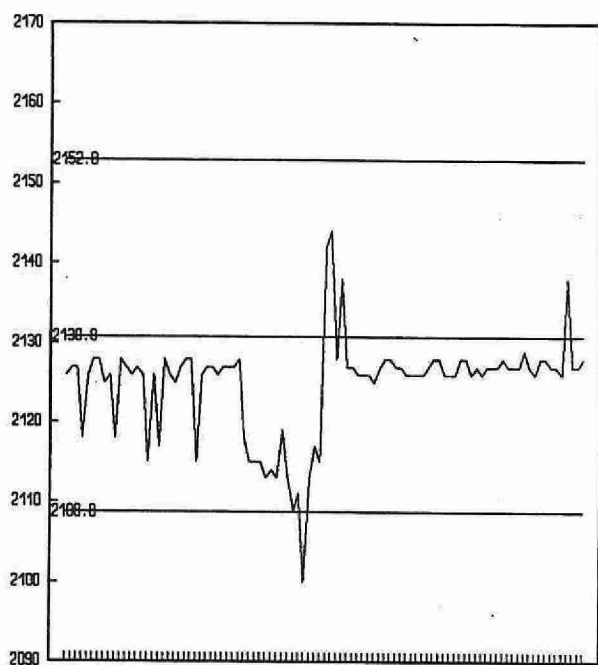
|        |   |        |     |     |
|--------|---|--------|-----|-----|
| 2108.6 | - | 2152.8 | for | A+B |
| 678.7  | - | 711.7  | for | A-B |
| 852.96 | - | 876.64 | for | C+D |
| 561.92 | - | 579.68 | for | C-D |

## DUPLICATES:

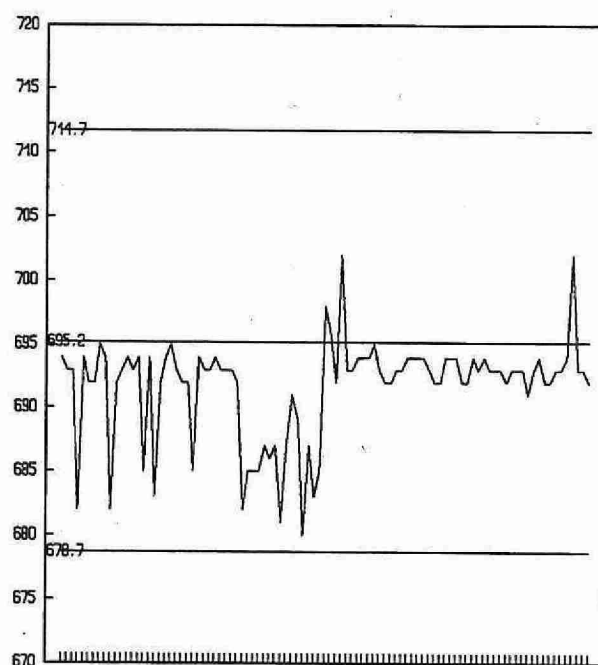
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 89              | 0 - 400                      | 1.2111                    | 0.7                            |
| 90              | 401 - 1000                   | 2.1344                    | 0.4                            |
| 31              | 1001 - 2000                  | 7.7418                    | 0.5                            |
| 13              | 2001 - 10000                 | 9.8058                    | 0.3                            |
| 223             | Overall                      | 1.7583                    |                                |

# CONDUCTIVITY ( $\mu\text{S}/\text{cm}$ )

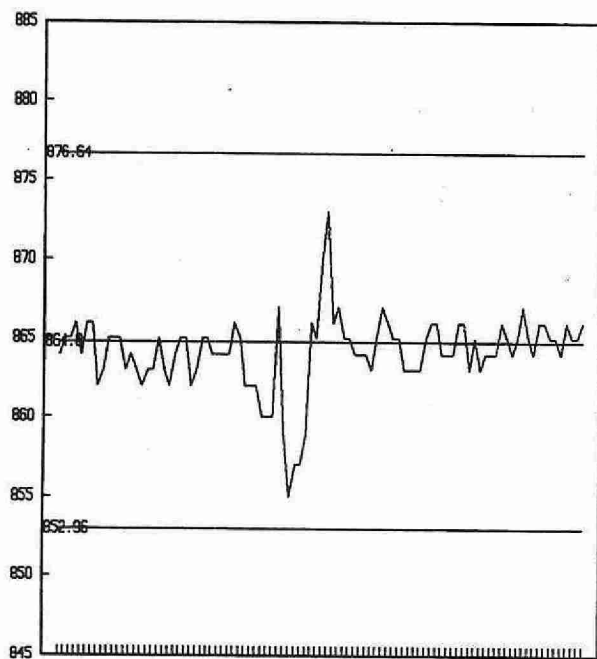
QUALITY CONTROL DATA FROM 04/01/94 TO 21/12/94



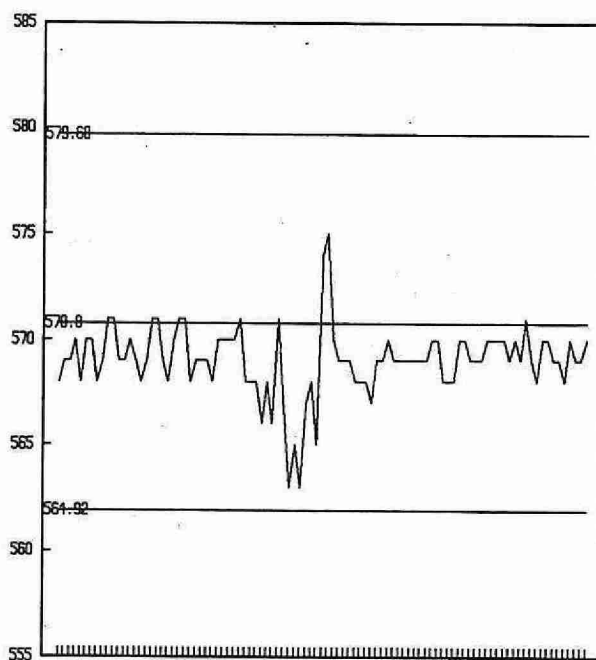
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

## CONDUCTIVITY

### IDENTIFICATION:

|                     |                    |                   |                                 |
|---------------------|--------------------|-------------------|---------------------------------|
| Laboratory Unit     | Titration          | Method Introduced | 20/05/87                        |
| Method Reference No | E3228A             | Units             | $\mu\text{S}/\text{cm}$ at 25°C |
| LIMS Product Code   | PHALK3228,COND3228 | Supervisor        | F. Lo                           |
| Sample Type/Matrix  | Landfill leachates |                   |                                 |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 75 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

After equilibration at 25°C, the conductivity of the sample is measured; samples are filtered first if necessary. Analysis is performed on supernatant or filtrate.

### INSTRUMENTATION:

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator.

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 5 | Current T value: 25 |
|--------------------------------|--------------------|---------------------|

### CONTROLS:

|             |                               |
|-------------|-------------------------------|
| Calibration | BL plus 4 standards, e.g. QCA |
|-------------|-------------------------------|

# CONDUCTIVITY

QUALITY CONTROL DATA FROM 22/02/94 TO 29/12/94

Laboratory Unit: Titration

Analytical Range: to 10000  $\mu\text{S}/\text{cm}$

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 22 | 6668                   | 6664               | -4        | 17.6363                |
| B:   | 22 | 2767                   | 2769               | 2         | 12.0694                |
| C:   | 22 | 1413                   | 1415               | 2         | 5.0965                 |
| D:   | 22 | 717                    | 718                | 1         | 2.5618                 |
| A+B: | 22 | 9435                   | 9433               | -2        | 26.2192                |
| A-B: | 22 | 3901                   | 3895               | -6        | 15.0324                |
| C+D: | 22 | 2130                   | 2133               | 3         | 7.1217                 |
| C-D: | 22 | 696                    | 697                | 1         | 3.7888                 |

s.d.(AB) S(between runs): 15.1

Sw(within run): 10.6

S/Sw: 1.4

s.d.(CD) S(between runs): 4.03

Sw(within run): 2.68

S/Sw: 1.5

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 9311 | - | 9559 | for | A+B |
| 3808 | - | 3994 | for | A-B |
| 2094 | - | 2167 | for | C+D |
| 668  | - | 724  | for | C-D |

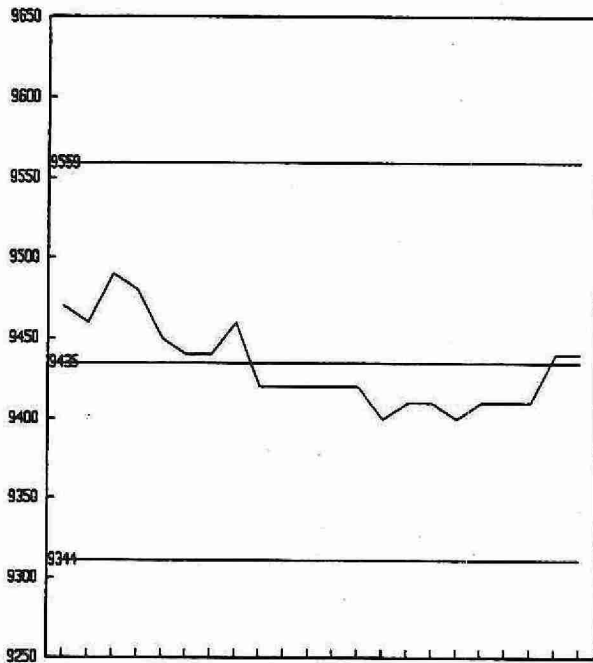
## DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 48           | 0 - 1000                  | 1.2306                 | 0.3                         |
| 6            | 1001 - 2000               | 1.3540                 | 0.1                         |
| 3            | 2001 - 10000              | 11.3373                | 0.2                         |
| 57           | Overall                   | 1.4430                 |                             |

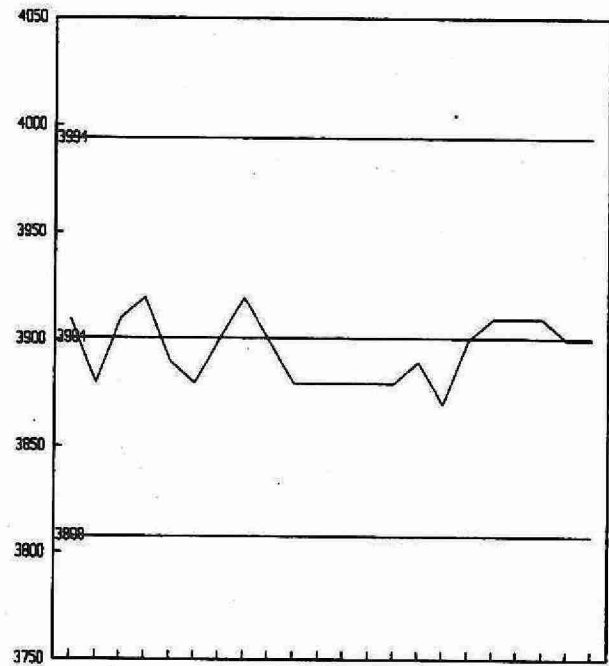


# CONDUCTIVITY ( $\mu\text{S}/\text{cm}$ )

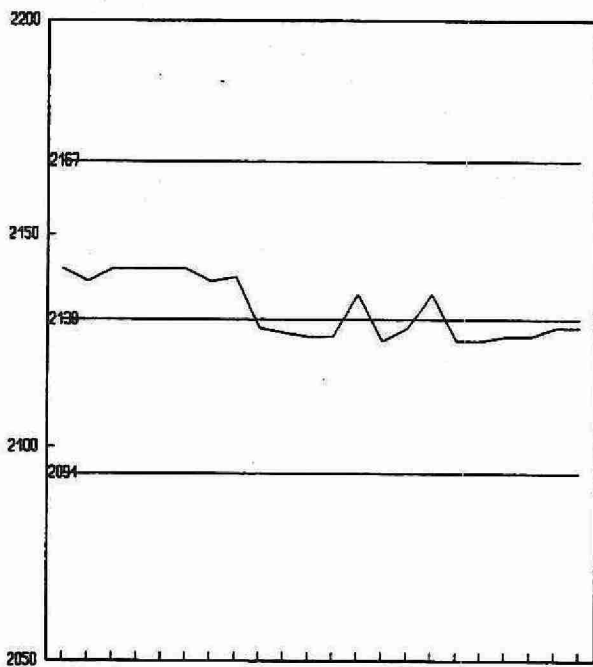
QUALITY CONTROL DATA FROM 22/02/94 TO 29/12/94



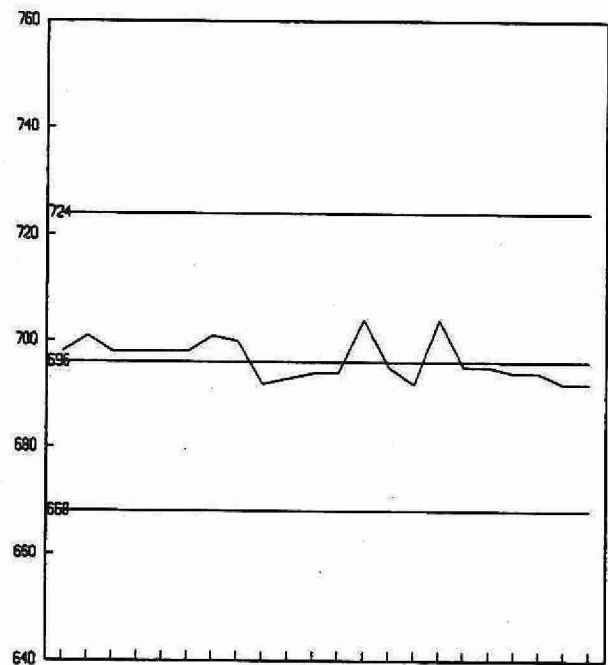
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

## CONDUCTIVITY

### IDENTIFICATION:

|                      |                       |                    |                                 |
|----------------------|-----------------------|--------------------|---------------------------------|
| Laboratory Unit:     | Titration             | Method Introduced: | 01/04/74                        |
| Method Reference No: | E3289A                | Units:             | $\mu\text{S}/\text{cm}$ at 25°C |
| LIMS Product Code:   | PHALCO3289,CONDPH3289 | Supervisor:        | F. Lo                           |
| Sample Type/Matrix:  | Rivers, Lakes         |                    |                                 |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: | 25 mL            |
| Container:         | Glass or plastic |

### ANALYTICAL PROCEDURE:

After equilibration at 25°C, the conductivity of the sample is measured.  
pH, Gran alkalinity and total fixed endpoint alkalinity are determined simultaneously.

### INSTRUMENTATION:

Automated modular continual flow conductivity system comprising of a sampler, water bath, pump, conductivity meter with cell plus microcomputer control and data processing software.

### REPORTING:

|                                |                    |                    |
|--------------------------------|--------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 1 | Current T value: 5 |
|--------------------------------|--------------------|--------------------|

### CONTROLS:

|              |  |
|--------------|--|
| Calibration: | BL plus 3 standards, e.g. QCA                                      |
| Drift:       | In run standards throughout the run (tap water diluted to 20% V/V) |

# CONDUCTIVITY

QUALITY CONTROL DATA FROM 06/01/94 TO 12/12/94

Laboratory Unit: Titration

Analytical Range: to 2000  $\mu\text{S}/\text{cm}$

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 87 | 717.8                  | 717.2              | -0.6      | 1.2903                 |
| B:   | 87 | 147.0                  | 147.7              | 0.7       | 0.5854                 |
| C:   | 87 | 147.0                  | 147.7              | 0.7       | 0.5854                 |
| D:   | 87 | 37.1                   | 37.8               | 0.7       | 0.4253                 |
| A+B: | 87 | 864.8                  | 864.9              | 0.1       | 1.5668                 |
| A-B: | 87 | 570.8                  | 569.5              | -1.3      | 1.2491                 |
| C+D: | 87 | 184.1                  | 185.5              | 1.4       | 0.7228                 |
| C-D: | 87 | 109.9                  | 110.0              | 0.1       | 0.7244                 |

s.d.(AB)    S(between runs): 1.00                      Sw(within run): 0.88                      S/Sw: 1.1

s.d.(CD)    S(between runs): 0.51                      Sw(within run): 0.51                      S/Sw: 1.0

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

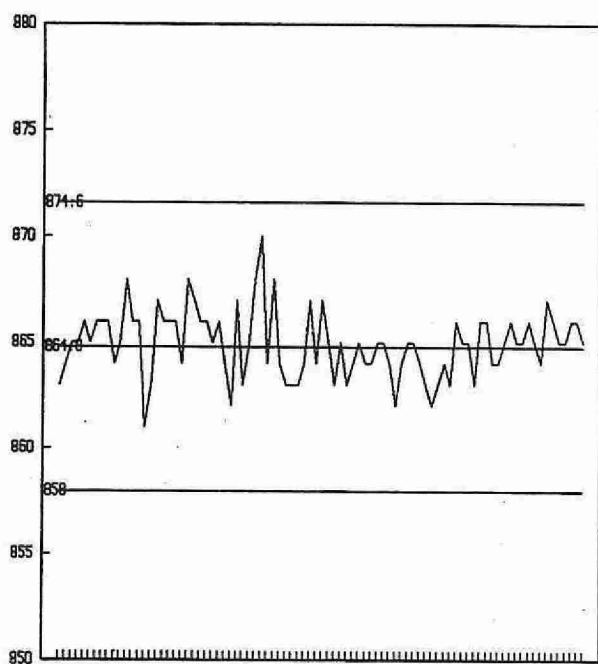
|        |   |        |     |     |
|--------|---|--------|-----|-----|
| 858    | - | 871.6  | for | A+B |
| 565.7  | - | 575.9  | for | A-B |
| 180.54 | - | 187.66 | for | C+D |
| 107.23 | - | 112.57 | for | C-D |

## DUPLICATES:

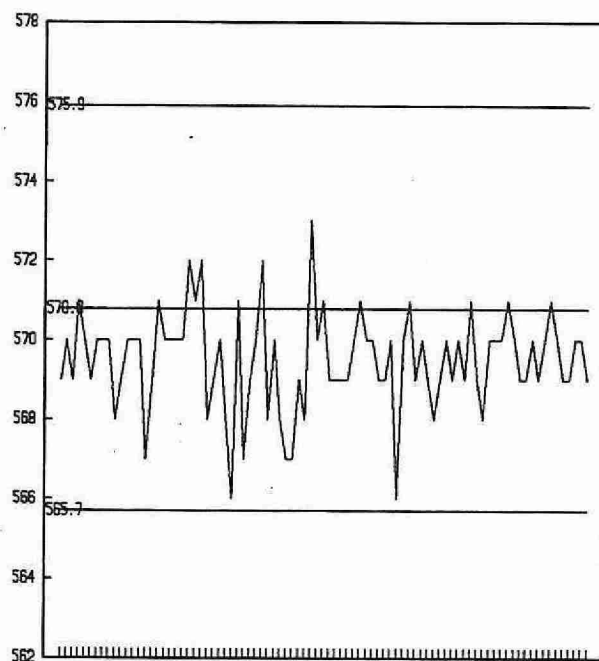
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 111          | 0 - 400                   | 1.0206                 | 0.5                         |
| 108          | 401 - 1000                | 1.7250                 | 0.3                         |
| 15           | 1001 - 2000               | 4.4081                 | 0.5                         |
| 11           | 2001 - 10000              | 7.2671                 | 0.2                         |
| 245          | Overall                   | 1.5030                 |                             |

# CONDUCTIVITY ( $\mu\text{S}/\text{cm}$ )

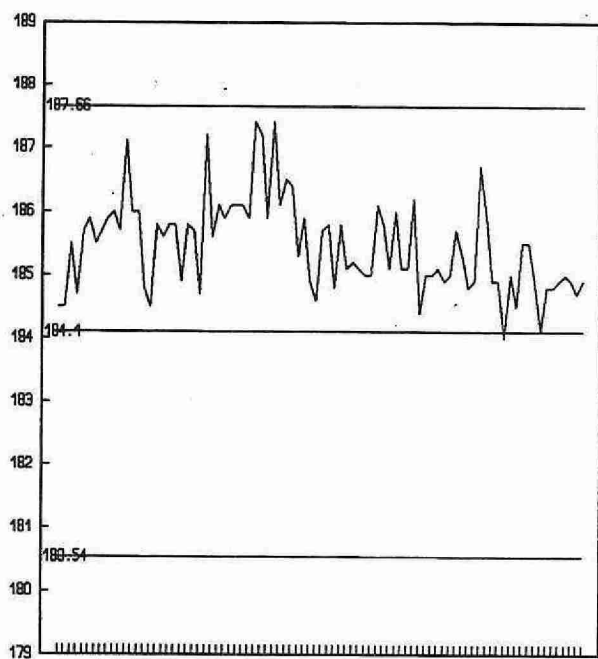
QUALITY CONTROL DATA FROM 06/01/94 TO 12/12/94



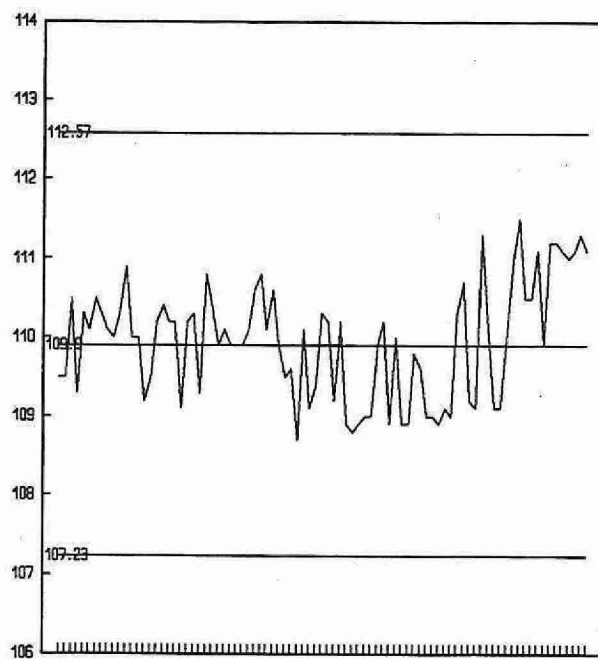
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

## COPPER, TOTAL

### IDENTIFICATION:

|                      |                               |                   |            |
|----------------------|-------------------------------|-------------------|------------|
| Laboratory Unit      | Dorset                        | Method Introduced | 01/01/91   |
| LIS Test Name Code   | CUUT                          | Units             | µg/L as Cu |
| Work Station Code    | DOTRACE                       | Unit Code         | 063829     |
| Method Code          | 005AF2                        | Supervisor        | J. McBride |
| Method Reference No. | E3376A                        |                   |            |
| Sample Type/Matrix   | Surface waters, precipitation |                   |            |

### SAMPLING:

|                   |  |
|-------------------|--|
| Quantity Required | 5 mL   |
| Container         | glass or plastic, capped, acidified to 0.25% with HNO <sub>3</sub> |

### ANALYTICAL PROCEDURE:

Samples are analyzed by GFAAS at 324.8nm.  
Absorbance : 0.8 at full scale

### INSTRUMENTATION:

A graphite furnace atomic absorption spectrometer with automated sampler.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.003 | Current T value: 0.015 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 4 standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | Long Term Blank, 1 NRC solution, 3 duplicates |
| Drift       | 1 blank plus 1 standard                       |

## COPPER, TOTAL

QUALITY CONTROL DATA FROM 21/03/94 TO 22/12/94

Laboratory Unit: Dorset

Full Scale: to 10 µg/L as Cu

### CALIBRATION CONTROL:

|     | n  | Mean<br>Concentration | Standard<br>Deviation (1) |
|-----|----|-----------------------|---------------------------|
| QCA | 49 | 0.5283                | 0.0283                    |
| NRC | 49 | 2.7487                | 0.0690                    |

### DUPLICATES:

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 151             | 0.00 - 1.00                  | 0.0690                    | 16.2                           |
| 25              | 1.01 - 2.00                  | 0.1069                    | 8.6                            |
| 6               | 2.01 - 5.00                  | 0.1601                    | 7.4                            |
| 1               | 5.01 - 10.0                  | N.A.                      | N.A.                           |
| 183             | Overall                      | 0.0785                    |                                |

### OTHER CHECKS:

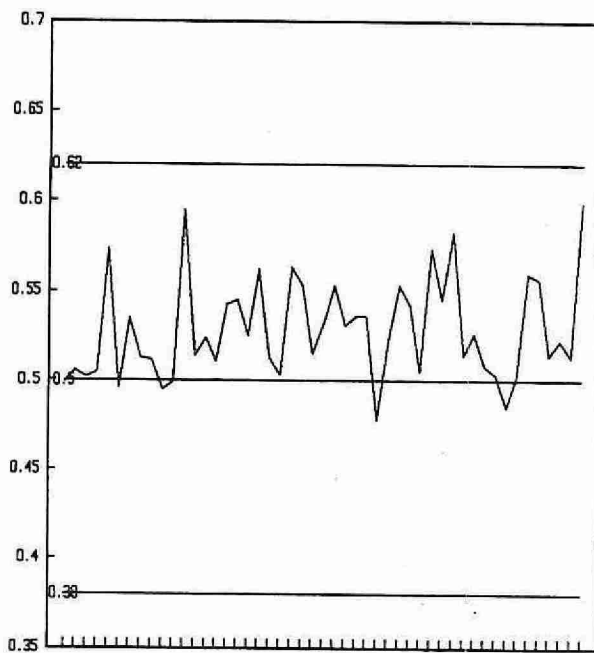
|     | n  | Mean   | Standard<br>Deviation (1) |
|-----|----|--------|---------------------------|
| NRC | 49 | 0.0192 | 0.0268                    |

### NOTES:

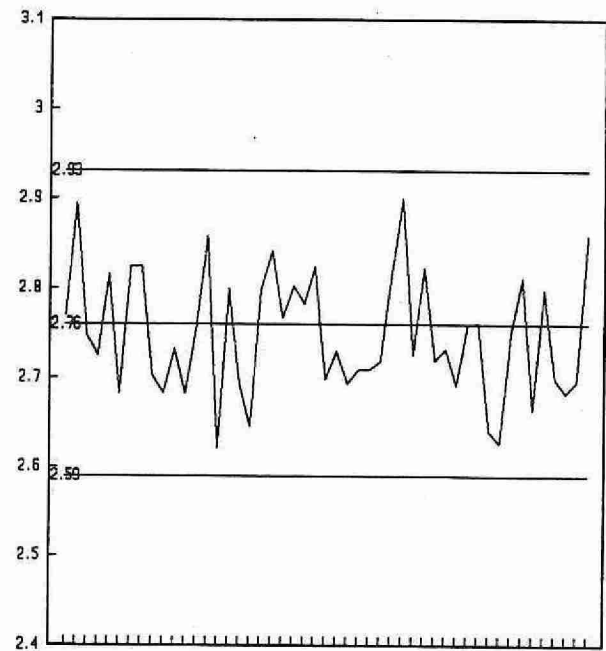
QCA is a low level calibration control standard prepared from an EPA ampoule.

# COPPER, TOTAL ( $\mu\text{g/L}$ )

QUALITY CONTROL DATA FROM 21/03/94 TO 22/12/94



QUALITY CONTROL STANDARD A



NRC REFERENCE SAMPLE

\_\_\_\_\_ CONTROL LIMIT

## CYANIDE, FREE

### IDENTIFICATION:

|                      |  |                    |             |
|----------------------|--|--------------------|-------------|
| Laboratory Unit:     | Colourimetry   | Method Introduced: |             |
| Method Reference No: | E3014A   | Units:             | mg/L        |
| LIMS Product Code:   | CN3014   | Supervisor:        | M. Rawlings |
| Sample Type/Matrix:  | Surface and drinking water, sewages and industrial wastes and in distillates from the manual distillation of samples for total cyanides. |                    |             |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: | 350 mL           |
| Container:         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Prescreen is conducted on all samples requiring Total Cyanide. If results of the prescreen are less than 0.01 mg/L CN<sup>-</sup> no further processing is required, otherwise the size of sample taken is judged upon the prescreen result. Free cyanide, includes the free, simple and weakly bound complex cyanides that decompose at 106°C, at pH 4. Cyanide is determined colourimetrically by the reaction of cyanide with chloramine-T to form cyanogen chloride which reacts with a combination of barbituric acid and isonicotinic acid to form a highly coloured coupling product which is measured at 600 nm.

### INSTRUMENTATION:

Distillation bath at 106°C.

Basic automated modular continuous flow system with colourimetric measurement through a 5 cm light path at 600 nm.

Data capture, reduction, and processing via a multistage microcomputer system.

### REPORTING:

|   |                        |                        |
|---|------------------------|------------------------|
| Maximum Significant Figures: 2 or the nearest W | Current W value: 0.001 | Current T value: 0.005 |
|---|------------------------|------------------------|

### CALIBRATION:

BL plus 1 standard

### CONTROLS:

|              |                           |
|--------------|---------------------------|
| Calibration: | 2 standards, e.g. QCA     |
| Drift:       | Blank and check standards |



# CYANIDE, FREE

QUALITY CONTROL DATA FROM 12/01/94 TO 29/12/94

Laboratory Unit: Colourimetry

Full Scale: to 0.2 mg/L as CN

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 63 | 0.15                      | 0.1476                | -0.0024   | 0.0058                    |
| B:   | 63 | 0.02                      | 0.0192                | -0.0008   | 0.0013                    |
| A+B: | 63 | 0.17                      | 0.1668                | -0.0032   | 0.0062                    |
| A-B: | 63 | 0.13                      | 0.1284                | -0.0016   | 0.0057                    |

s.d.(AB) S(between runs): 0.0042 Sw(within run): 0.0040 S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

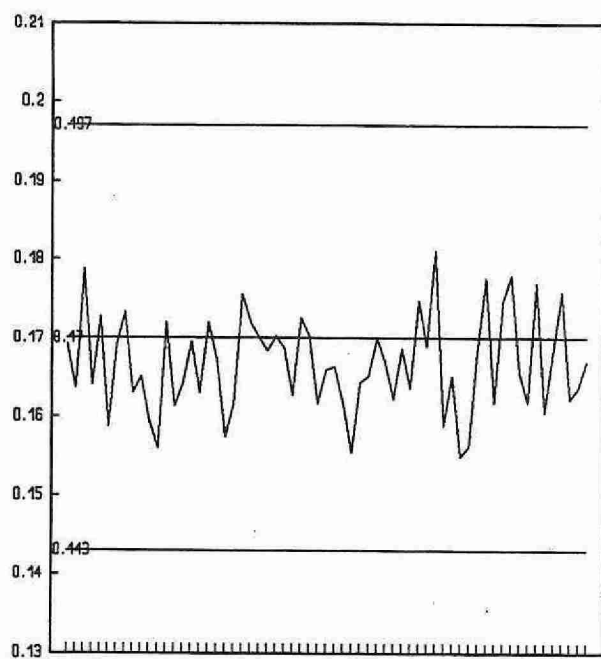
0.1430 - 0.1970 for A+B  
0.1105 - 0.1495 for A-B

## DUPLICATES:

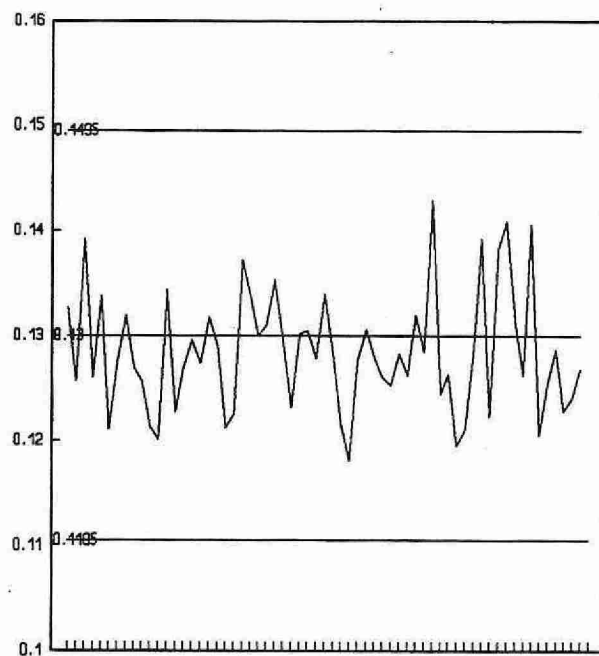
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 71              | 0.00 - 0.02                  | 0.0004                    | 30.9                           |
| 5               | 0.02 - 0.04                  | 0.0012                    | 3.5                            |
| 7               | 0.04 - 0.10                  | 0.0012                    | 2.4                            |
| 2               | 0.10 - 0.20                  | n.a.                      | n.a.                           |
| 85              | Overall                      | 0.0006                    | 8.8                            |

CYANIDE, FREE (mg/L as CN)

QUALITY CONTROL DATA FROM 12/01/94 TO 29/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## CYANIDE

### IDENTIFICATION:

|                      |  |                    |             |
|----------------------|--|--------------------|-------------|
| Laboratory Unit:     | Colourimetry   | Method Introduced: |             |
| Method Reference No: | E3015A   | Units:             | mg/L        |
| LIMS Product Code:   | CN3015   | Supervisor:        | M. Rawlings |
| Sample Type/Matrix:  | Surface and drinking water, sewage, landfill leachates, and industrial wastes. |                    |             |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: |                  |
| Container:         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Prescreen is conducted on all samples requiring Total Cyanide through the automated distillation procedure. Total Cyanides, including free, simple and complex cyanides are distilled out of a tartaric acid reflux/distillation as HCN and trapped in an alkaline solution. Cyanide is determined colourimetrically by the reaction of cyanide with chloramine-T to form cyanogen chloride which reacts with a combination of barbituric acid and isonicotinic acid to form a highly coloured coupling product which is measured at 600 nm.

### INSTRUMENTATION:

Distillation bath.

Basic automated modular continuous flow system with colourimetric measurement through a 5 cm light path at 600 nm.

Data capture, reduction, and processing via a multistage microcomputer system.

### REPORTING:

|   |                        |                        |
|---|------------------------|------------------------|
| Maximum Significant Figures: 2 or the nearest W | Current W value: 0.001 | Current T value: 0.005 |
|---|------------------------|------------------------|

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

|              |                        |
|--------------|------------------------|
| Calibration: | 2 standards, e.g. QCA  |
| Drift:       | BL and check standards |

# CYANIDE, TOTAL

QUALITY CONTROL DATA FROM 12/01/94 TO 29/12/94

Laboratory Unit: Colourimetry

Full Scale: to 0.2 mg/L as CN

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 69 | 0.15                   | 0.1457             | -0.0043   | 0.0041                 |
| B:   | 69 | 0.02                   | 0.0185             | -0.0015   | 0.0010                 |
| A+B: | 69 | 0.17                   | 0.1642             | -0.0058   | 0.0044                 |
| A-B: | 69 | 0.13                   | 0.1273             | -0.0027   | 0.0040                 |

s.d.(AB) S(between runs): 0.0030

Sw(within run): 0.0028

S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.1500 - 0.1900 for A+B

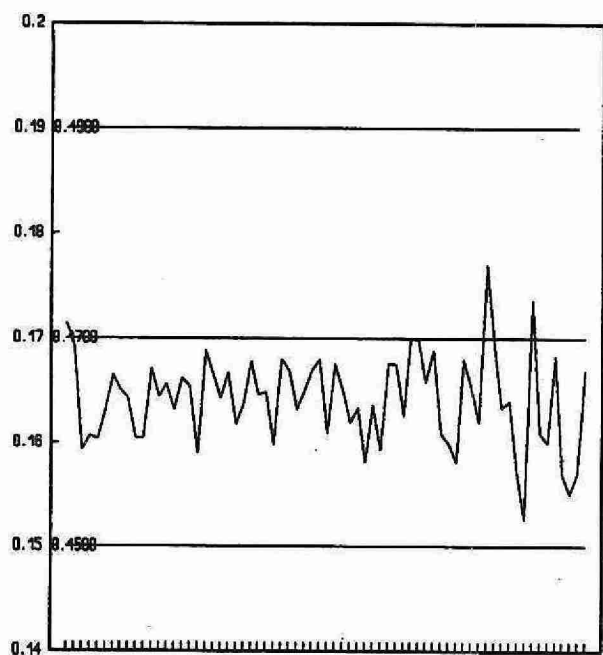
0.1172 - 0.1428 for A-B

## DUPLICATES:

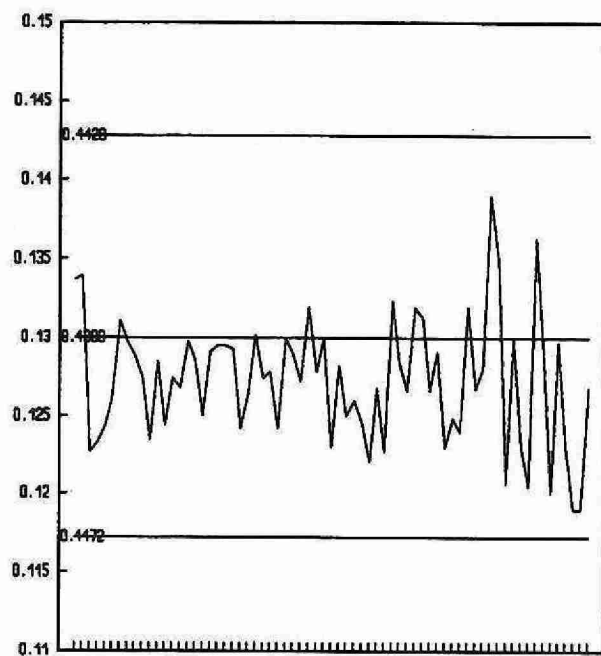
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 43           | 0.00 - 0.02               | 0.0008                 | 55.1                        |
| 20           | 0.02 - 0.04               | 0.0005                 | 20.5                        |
| 30           | 0.04 - 0.10               | 0.0006                 | 16.6                        |
| 0            | 0.10 - 0.20               | n.a.                   | n.a.                        |
| 93           | Overall                   | 0.0005                 | 25.0                        |

CYANIDE, TOTAL (mg/L as CN)

QUALITY CONTROL DATA FROM 12/01/94 TO 29/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## FLUORIDE

### IDENTIFICATION:

|                      |                                   |                   |            |
|----------------------|-----------------------------------|-------------------|------------|
| Laboratory Unit      | Dorset                            | Method Introduced | 01/06/80   |
| LIS Test Name Code   | FFIDUR                            | Units             | µg/L as F  |
| Work Station Code    | DOSPF                             | Unit Code         | 063809     |
| Method Code          | 001AIE                            | Supervisor        | J. McBride |
| Method Reference No. | E3041A                            |                   |            |
| Sample Type/Matrix   | Precipitation, Lakes, and Streams |                   |            |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 50 mL   |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Fluoride is determined by specific ion electrode using an automated flow system. Prior to measurement the sample is mixed with a high ionic strength buffer containing; sodium citrate, disodium ethylenediaminetetraacetate (EDTA), phosphoric acid, and sufficient sodium hydroxide to obtain pH 6.7.

### INSTRUMENTATION:

Automated modular continuous flow ion specific electrode system.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|              |  |
|--------------|--|
| Calibration  | 2 standards, e.g. QCA  |
| Drift        | BL plus 1 standard in duplicate  |
| Interference | Combined fluoride and aluminum standard confirms that aluminum is not an interference. |

### NOTES:

Values for recoveries are based upon the average recovery value obtained.

# FLUORIDE

QUALITY CONTROL DATA FROM 12/01/94 TO 21/12/94

Laboratory Unit: Dorset

Full Scale: to 70.0 µg/L as F

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 28 | 48                     | 48.60              | 0.60      | 0.6194                 |
| B:   | 28 | 24                     | 24.30              | 0.30      | 0.7341                 |
| A+B: | 28 | 72                     | 72.68              | 0.68      | 1.2736                 |
| A-B: | 28 | 24                     | 24.30              | 0.30      | 0.7574                 |

s.d.(AB) S(between runs): 0.68

Sw(within run): 0.54

S/Sw: 1.27

The calibration is accepted if the calibration control values obtained lie within the ranges:

67.5 - 76.5 for A+B

21 - 27 for A-B

## DUPLICATES:

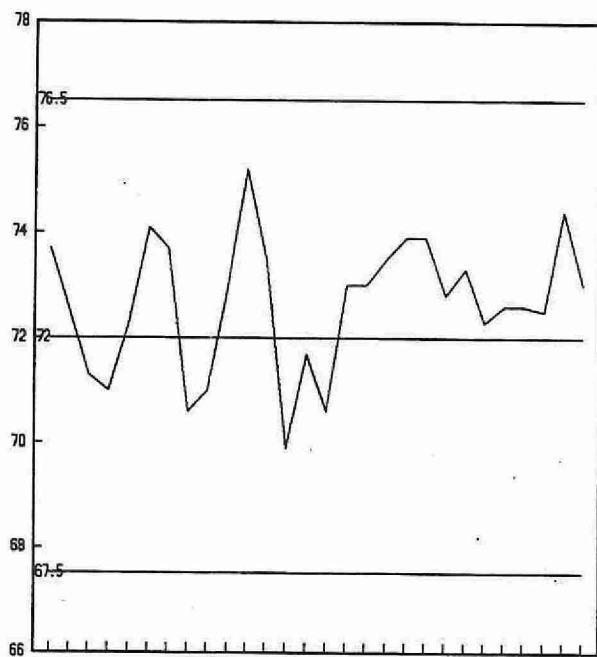
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 23           | 0.00 - 7.00               | 0.2655                 | 1.6                         |
| 2            | 7.01 - 14.00              | N.A.                   | N.A.                        |
| 24           | 14.01 - 53.00             | 0.6619                 | 0.5                         |
| 51           | 35.01 - 70.00             | 1.1797                 | 4.6                         |
| 100          | Overall                   | 0.8711                 |                             |

## OTHER CHECKS:

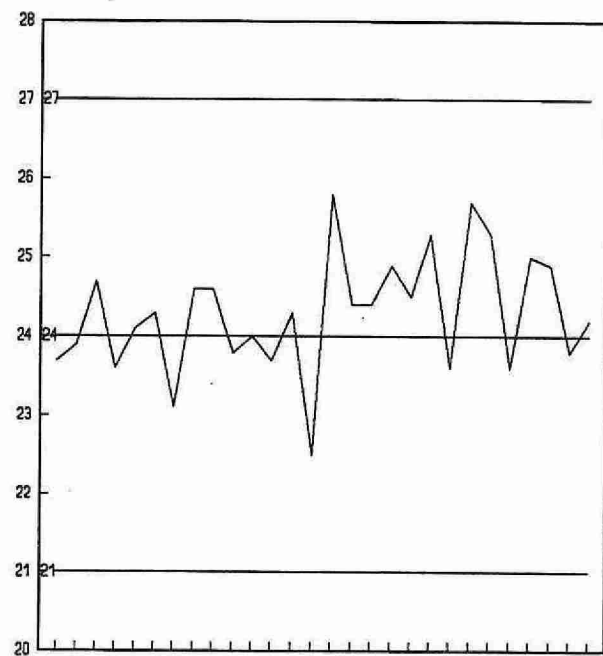
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Al Interference | 28 | 59.296 | 1.4458                 |

**FLUORIDE** ( $\mu\text{g/L}$  as F)

QUALITY CONTROL DATA FROM 12/01/94 TO 21/12/94



QUALITY CONTROL SAMPLE A+B



QUALITY CONTROL SAMPLE A-B

CONTROL LIMIT



## FLUORIDE

### IDENTIFICATION:

|                     |   |                   |             |
|---------------------|---|-------------------|-------------|
| Laboratory Unit     | Colourimetry  | Method Introduced | before '74  |
| Method Reference No | E3369A  | Units             | mg/L as F   |
| LIMS Product Code   | FNOT3369  | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Domestic Waters, Surface Waters, Leachates, Effluents |                   |             |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 50 mL   |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Using an automated flow system the sample is distilled in the presence of sulphuric acid at 160°C; the distillate is then reacted (in an acetic acid-acetate buffer media) with Alizarin Fluorine Blue and lanthanum nitrate to form a ternary Alizarin Blue-lanthanide-fluoride complex.  
Approximate absorbance: 0.8 at the full scale level.

### INSTRUMENTATION:

Modular continuous flow colourimetric system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.01 | Current T value: 0.05 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 6 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTB plus 3 standards, e.g. QCA                 |
| Drift       | BL every 10 samples; standard every 20 samples |

# FLUORIDE

QUALITY CONTROL DATA FROM 05/01/94 TO 22/12/94

Laboratory Unit: Colourimetry

Analytical Range: to 2.0 mg/L as F

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 75 | 1.6                    | 1.605              | -0.005    | 0.0210                 |
| B:   | 75 | 0.8                    | 0.794              | 0.006     | 0.0167                 |
| C:   | 75 | 0.16                   | 0.162              | -0.002    | 0.0093                 |
| A+B: | 75 | 2.4                    | 2.399              | 0.001     | 0.0288                 |
| A-B: | 75 | 0.8                    | 0.812              | -0.012    | 0.0248                 |
| B+C: | 75 | 0.96                   | 0.955              | 0.005     | 0.0205                 |
| B-C: | 75 | 0.64                   | 0.632              | 0.008     | 0.0177                 |

s.d.(AB) S(between runs): 0.019 Sw(within run): 0.018 S/Sw: 1.1

s.d.(BC) S(between runs): 0.013 Sw(within run): 0.013 S/Sw: 1.1

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |         |
|------|---|------|---------|
| 2.30 | - | 2.50 | for A+B |
| 0.73 | - | 0.87 | for A-B |
| 0.90 | - | 1.00 | for B+C |
| 0.60 | - | 0.68 | for B-C |

## DUPLICATES:

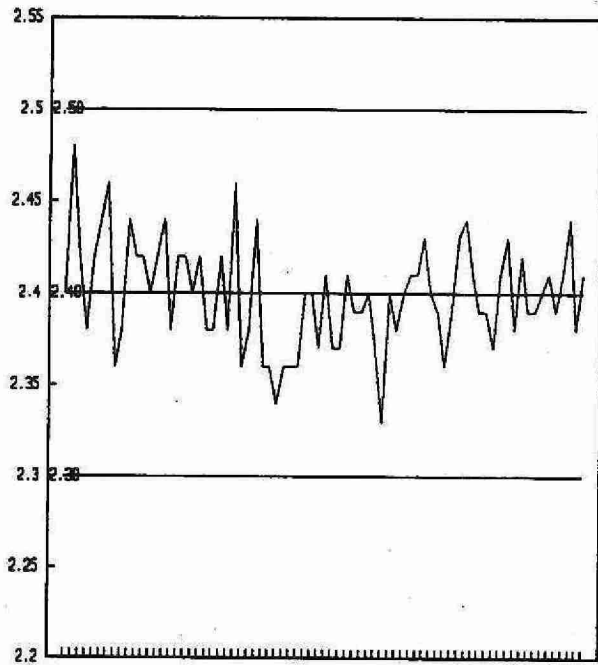
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 157          | 0.00 - 0.20               | 0.0111                 | 11.7                        |
| 15           | 0.21 - 0.40               | 0.0109                 | 3.9                         |
| 29           | 0.41 - 1.00               | 0.0199                 | 2.8                         |
| 18           | 1.01 - 2.00               | 0.0251                 | 1.9                         |
| 219          | Overall                   | 0.0129                 |                             |

## OTHER CHECKS:

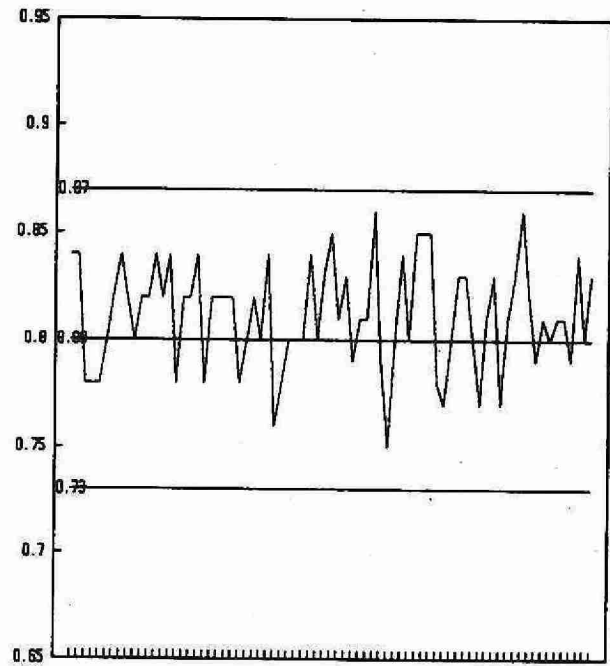
|                 | n  | Data Mean | Standard (1) Deviation |
|-----------------|----|-----------|------------------------|
| Long Term Blank | 75 | -0.0039   | 0.0133                 |

FLUORIDE (mg/L as F)

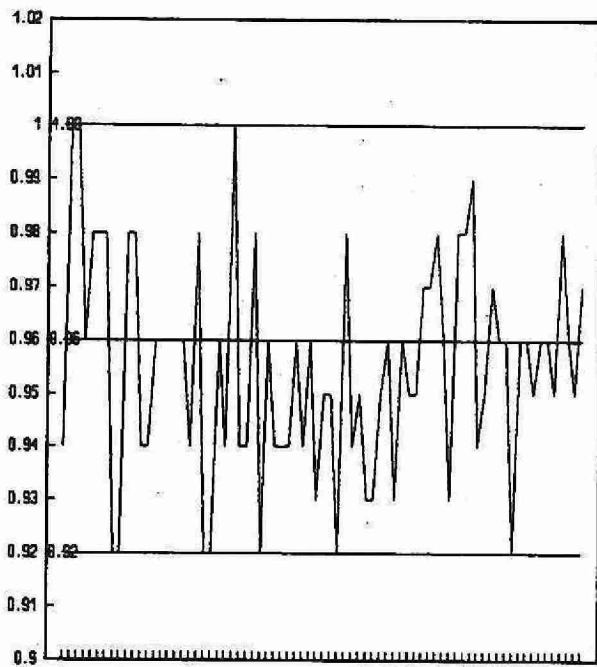
QUALITY CONTROL DATA FROM 05/01/94 TO 22/12/94



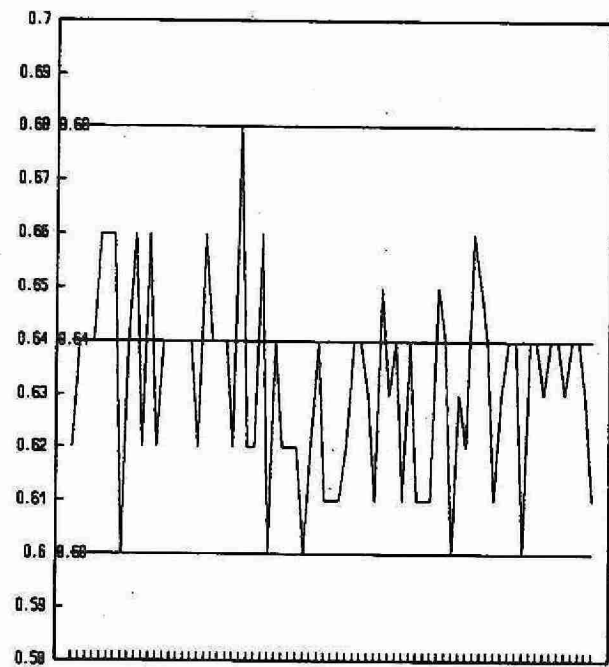
QUALITY CONTROL SAMPLE A+B



QUALITY CONTROL SAMPLE A-B



QUALITY CONTROL SAMPLE B+C



QUALITY CONTROL SAMPLE B-C

CONTROL LIMIT

## HARDNESS

### IDENTIFICATION:

|                      |                              |                   |                           |
|----------------------|------------------------------|-------------------|---------------------------|
| Laboratory Unit      | Atomic Absorption            | Method Introduced | 01/04/74                  |
| Method Reference No. | E3171A                       | Units             | mg/L as CaCO <sub>3</sub> |
| LIMS Product Code    | CAT3171,HARD3171             | Supervisor        | J. McBride                |
| Sample Type/Matrix   | Rivers, Lakes, Soil Extracts |                   |                           |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analysed for calcium and magnesium by AAS (E3171A). Hardness is calculated using the formula:

$$HARDT = (CAUR \times 2.497) + (MGUR \times 4.118)$$

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

Refer to Calcium and Magnesium tests (E3171A)

### CONTROLS:

Refer to Calcium and Magnesium tests (E3171A)

## HARDNESS

### IDENTIFICATION:

|                      |  |                   |                           |
|----------------------|--|-------------------|---------------------------|
| Laboratory Unit      | Atomic Absorption  | Method Introduced | 08/04/86                  |
| Method Reference No. | E3217A   | Units             | mg/L as CaCO <sub>3</sub> |
| LIMS Product Code    | CAT3217,CATS3217,HARD3217  | Supervisor        | J. McBride                |
| Sample Type/Matrix   | Domestic Waters, Leachates, Effluents, Sewage, Industrial Wastes |                   |                           |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analysed for calcium and magnesium by AAS (E3217A). Hardness is calculated using the formula:

$$HARDT = (CAUR \times 2.497) + (MGUR \times 4.118)$$

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |
|--------------------------------|----------------------|----------------------|

### CALIBRATION:

Refer to Calcium and Magnesium tests (E3217A)

### CONTROLS:

Refer to Calcium and Magnesium tests (E3217A)

## HARDNESS

### IDENTIFICATION:

|                      |                |                   |                           |
|----------------------|----------------|-------------------|---------------------------|
| Laboratory Unit      | Dorset         | Method Introduced | 01/04/74                  |
| LIS Test Name Code   | HARDT          | Units             | mg/L as CaCO <sub>3</sub> |
| Work Station Code    | DOFLAME        | Unit Code         | 064915                    |
| Method Code          | CALC10         | Supervisor        | J. McBride                |
| Method Reference No. | E3249A         |                   |                           |
| Sample Type/Matrix   | Rivers, Lakes, |                   |                           |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analysed for calcium and magnesium by AAS at workstation DOFLAME. Hardness is calculated using the formula:

$$HARDT = (CAUR \times 2.497) + (MGUR \times 4.118)$$

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

Refer to Calcium and Magnesium tests at DOFLAME

### CONTROLS:

Refer to Calcium and Magnesium tests at DOFLAME

## IRON, TOTAL

### IDENTIFICATION:

|                      |  |                   |            |
|----------------------|--|-------------------|------------|
| Laboratory Unit      | Dorset                                       | Method Introduced | 20/07/88   |
| LIS Test Name Code   | FEUT   | Units             | µg/L as Fe |
| Work Station Code    | DOFEMN                                       | Unit Code         | 063826     |
| Method Code          | 504BC2                                       | Supervisor        | J. McBride |
| Method Reference No. | E3303B                                       |                   |            |
| Sample Type/Matrix   | Surface water, precipitation, soil leachates |                   |            |

### SAMPLING:

|                   |  |
|-------------------|--|
| Quantity Required | 25 mL  |
| Container         | Glass or plastic, capped, acidified to 0.25% with HNO <sub>3</sub> |

### ANALYTICAL PROCEDURE:

An undigested sample is introduced to an in-line UV digester. A reducing agent and a buffer are added to the sample. TPTZ is added to develop a blue colour, the intensity of which is proportional to the concentration of Fe in the sample. The colour is measured at 600nm.

### INSTRUMENTATION:

- An AAII autoanalyzer with colorimeter and automated sampler.

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |
|--------------------------------|--------------------|---------------------|

### CALIBRATION:

BL plus 4 standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | Long Term blank, 3 QC's, 4 duplicates   |
| Drift       | Blank plus 1 standard every 10 samples. |

# IRON, TOTAL

QUALITY CONTROL DATA FROM 10/01/94 TO 20/12/94

Laboratory Unit: Dorset

Full Scale: to 1000.0 µg/L as Fe

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 40 | 750.0                  | 751.23             | 1.23      | 4.5993                 |
| B:   | 40 | 250.0                  | 247.45             | -2.55     | 3.7345                 |
| C:   | 40 | 50.0                   | 48.30              | -1.70     | 2.0280                 |
| A+B: | 40 | 1000.0                 | 999.08             | -0.93     | 5.6131                 |
| A-B: | 40 | 500.0                  | 503.55             | 3.55      | 6.1392                 |
| B+C: | 40 | 300.0                  | 295.90             | -4.10     | 4.8664                 |
| B-C: | 40 | 200.0                  | 199.40             | -0.60     | 4.4538                 |

s.d.(AB) S(between runs): 4.19

Sw(within run): 4.34

S/Sw: 0.97

s.d.(BC) S(between runs): 3.00

Sw(within run): 3.15

S/Sw: 0.95

The calibration is accepted if the calibration control values obtained lie within the ranges:

|     |   |      |     |     |
|-----|---|------|-----|-----|
| 975 | - | 1025 | for | A+B |
| 520 | - | 480  | for | A-B |
| 285 | - | 315  | for | B+C |
| 190 | - | 210  | for | B-C |

## DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 84           | 0 - 100                   | 1.9732                 | 7.0                         |
| 29           | 101 - 200                 | 4.2481                 | 2.7                         |
| 25           | 201 - 500                 | 5.1001                 | 1.6                         |
| 15           | 501 - 1000                | 11.2881                | 1.4                         |
| 153          | Overall                   | 3.3422                 |                             |

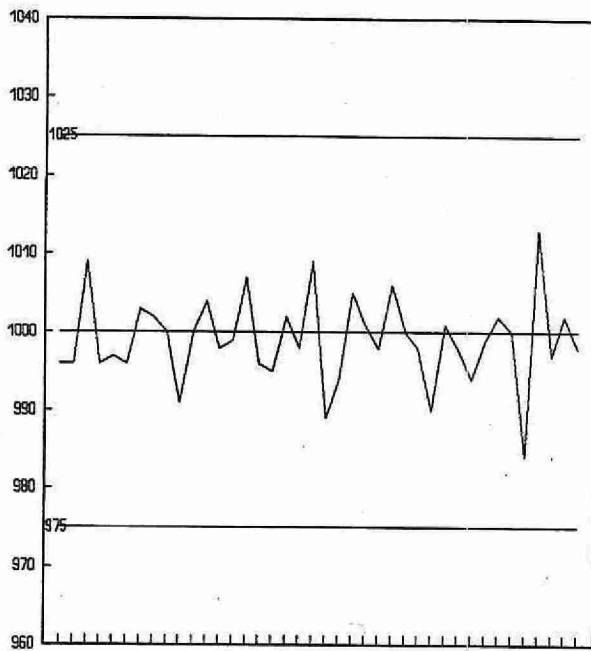
## OTHER CHECKS:

|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 40 | -0.200 | 1.1810                 |

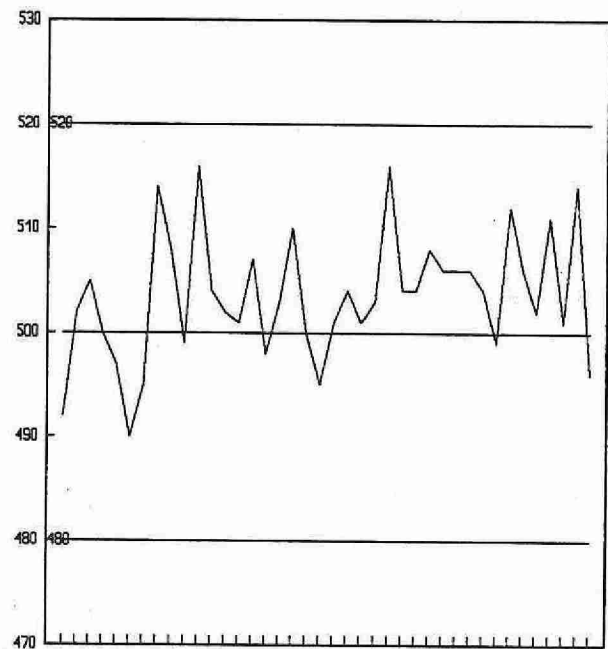


# IRON, TOTAL ( $\mu\text{g/L}$ as Fe)

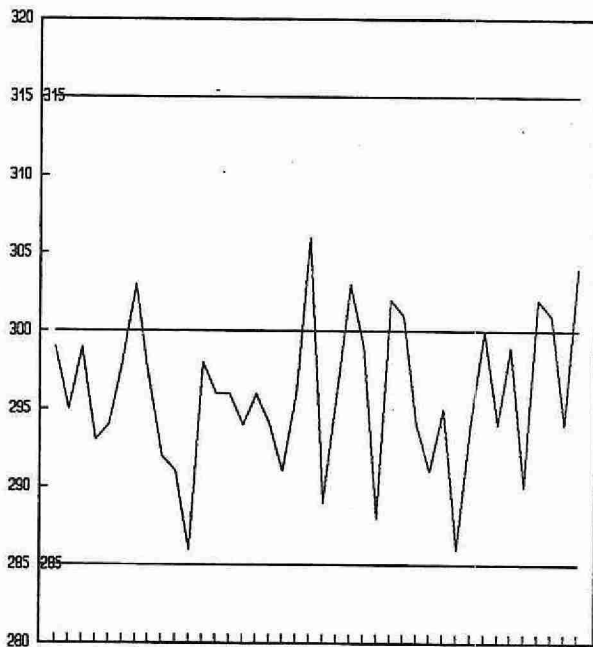
QUALITY CONTROL DATA FROM 10/01/94 TO 20/12/94



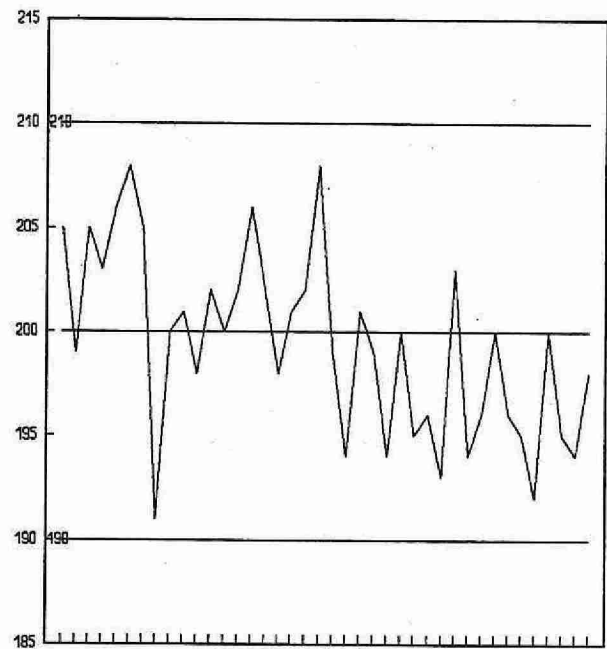
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

**LEAD, TOTAL****IDENTIFICATION:**

|                      |                               |                   |            |
|----------------------|-------------------------------|-------------------|------------|
| Laboratory Unit      | Dorset                        | Method Introduced | 01/01/91   |
| LIS Test Name Code   | PBUT                          | Units             | µg/L as Pb |
| Work Station Code    | DOTRACE                       | Unit Code         | 063882     |
| Method Code          | 005AF2                        | Supervisor        | J. McBride |
| Method Reference No. | E3376A                        |                   |            |
| Sample Type/Matrix   | Surface waters, precipitation |                   |            |

**SAMPLING:**

|                   |  |
|-------------------|--|
| Quantity Required | 5 mL   |
| Container         | Glass or plastic, capped, acidified to 0.25% with HNO <sub>3</sub> |

**ANALYTICAL PROCEDURE:**

Samples are analyzed by GFAAS at 217nm.

Absorbance : 0.35 at full scale

**INSTRUMENTATION:**

A graphite furnace atomic absorption spectrometer with automated sampler.

**REPORTING:**

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.003 | Current T value: 0.015 |
|--------------------------------|------------------------|------------------------|

**CALIBRATION:**

BL plus 5 standards

**CONTROLS:**

|             |   |
|-------------|---|
| Calibration | Long Term Blank, 1 NRC solution, 3 duplicates |
| Drift       | 1 blank plus 1 standard                       |

# LEAD, TOTAL

QUALITY CONTROL DATA FROM 17/03/94 TO 22/12/94

Laboratory Unit: Dorset

Full Scale: to 10 µg/L as Pb

## CALIBRATION CONTROL:

|     | n  | Mean<br>Concentration | Standard<br>Deviation (1) |
|-----|----|-----------------------|---------------------------|
| QCA | 26 | 0.5203                | 0.0375                    |
| NRC | 26 | 0.1250                | 0.0042                    |

## DUPLICATES:

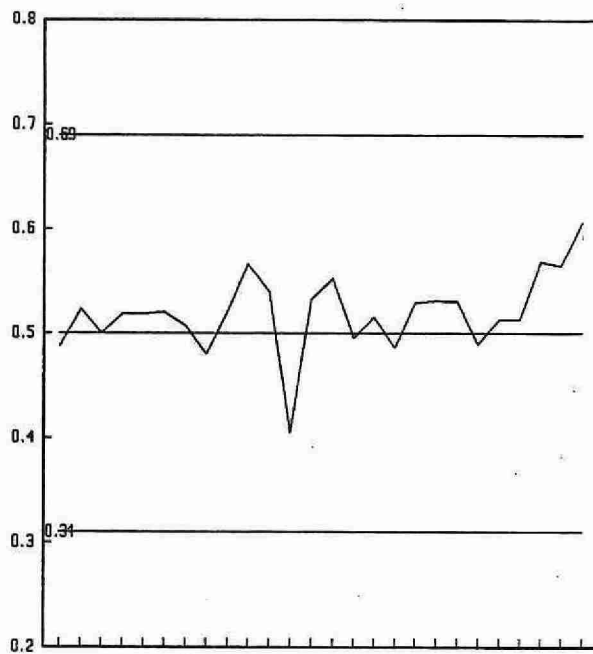
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 73              | 0.00 - 1.00                  | 0.0155                    | 36.4                           |
| 4               | 1.01 - 2.00                  | 0.0872                    | 7.8                            |
| 1               | 2.01 - 5.00                  | N.A.                      | N.A.                           |
| 0               | 5.01 - 10.0                  | N.A.                      | N.A.                           |
| 78              | Overall                      | 0.0173                    |                                |

## NOTES:

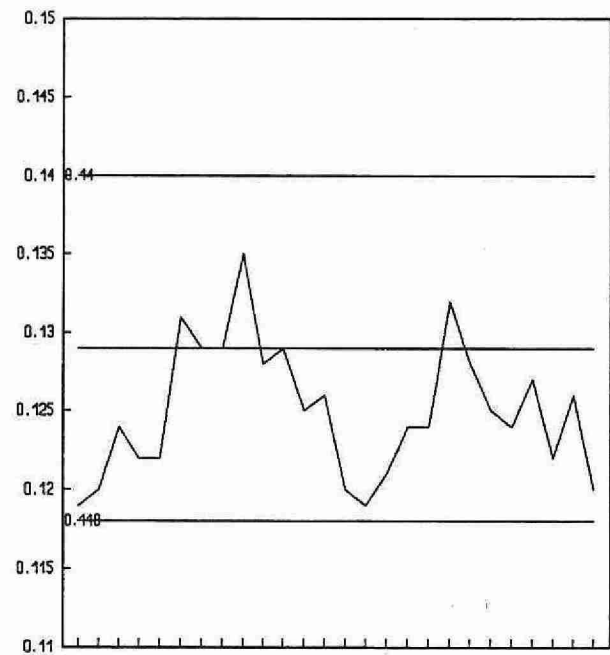
QCA is a low level calibration control standard prepared from an EPA ampoule.

**LEAD, TOTAL** ( $\mu\text{g/L}$ )

QUALITY CONTROL DATA FROM 17/03/94 TO 12/12/94



QUALITY CONTROL STANDARD A



NRC REFERENCE SAMPLE

CONTROL LIMIT

## MAGNESIUM

### IDENTIFICATION:

|                      |                   |                   |            |
|----------------------|-------------------|-------------------|------------|
| Laboratory Unit      | Atomic Absorption | Method Introduced | 18/05/79   |
| Method Reference No. | E3146A            | Units             | mg/L as Mg |
| LIMS Product Code    | CAT3146           | Supervisor        | J. McBride |
| Sample Type/Matrix   | Precipitation     |                   |            |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 5 mL    |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.001 | Current T value: 0.005 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 2 standards, e.g., QCA       |
| Drift       | BL, reslope standard every 10 samples. |

# MAGNESIUM

QUALITY CONTROL DATA FROM 03/02/94 TO 15/12/94

Laboratory Unit: Atomic Absorption

Full Scale: to 0.500 mg/L as Mg

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 30 | 0.30                   | 0.3022             | 0.0022    | 0.0032                 |
| B:   | 30 | 0.05                   | 0.0505             | 0.0005    | 0.0009                 |
| A+B: | 30 | 0.35                   | 0.3527             | 0.0027    | 0.0038                 |
| A-B: | 30 | 0.25                   | 0.2516             | 0.0016    | 0.0027                 |

s.d.(AB)

S(between runs): 0.0023

Sw(within run): 0.0019

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.338 - 0.362 for A+B

0.241 - 0.259 for A-B

## DUPLICATES:

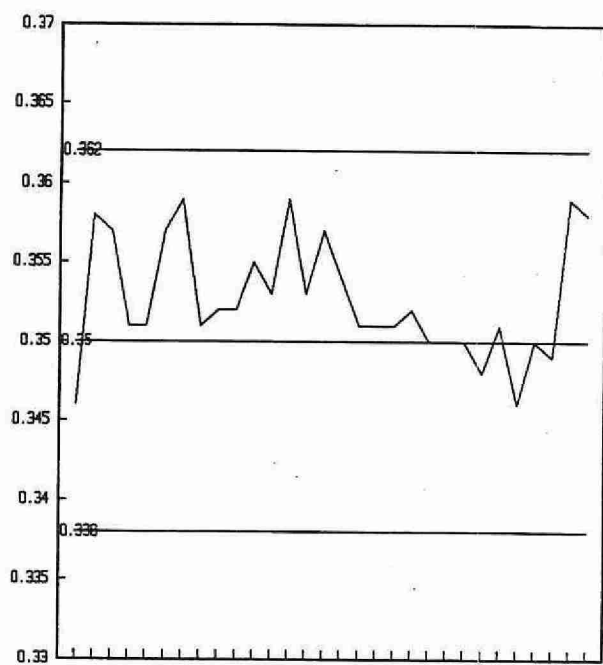
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 45           | 0.000 - 0.050             | 0.0007                 | 3.6                         |
| 10           | 0.051 - 0.100             | 0.0004                 | 0.5                         |
| 5            | 0.101 - 0.250             | 0.0004                 | 0.3                         |
| 10           | 0.250 - 0.500             | 0.0022                 | 0.6                         |
| 70           | OVERALL                   | 0.0009                 |                             |

## OTHER CHECKS:

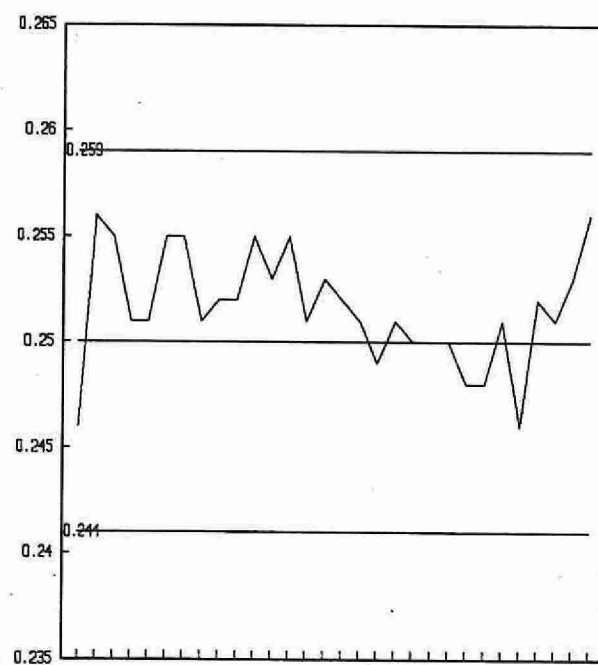
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 30 | 0.0006 | 0.0019                 |

**MAGNESIUM** (mg/L as Mg)

QUALITY CONTROL DATA FROM 03/02/94 TO 15/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

\_\_\_\_\_ CONTROL LIMIT

## MAGNESIUM

### IDENTIFICATION:

|                      |                                      |                   |            |
|----------------------|--------------------------------------|-------------------|------------|
| Laboratory Unit      | Atomic Absorption                    | Method Introduced | 01/04/74   |
| Method Reference No. | E3171A                               | Units             | mg/L as Mg |
| LIMS Product Code    | CAT3171,MG3171,HARD3171              | Supervisor        | J. McBride |
| Sample Type/Matrix   | Surface Waters, DWSP Drinking Waters |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.19 at the full scale level.

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                       |                      |
|--------------------------------|-----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.1 |
|--------------------------------|-----------------------|----------------------|

### CALIBRATION:

BL plus 11 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA                   |
| Drift       | BL every 10 samples; 2 standards every 20 samples. |



# MAGNESIUM

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94

Laboratory Unit: Atomic Absorption

Full Scale: to 10.0 mg/L as Mg

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 164 | 8.00                   | 8.04               | 0.04      | 0.0653                 |
| B:   | 164 | 2.00                   | 2.02               | 0.02      | 0.0229                 |
| C:   | 164 | 0.50                   | 0.506              | 0.006     | 0.0071                 |
| A+B: | 164 | 10.0                   | 10.06              | 0.06      | 0.0757                 |
| A-B: | 164 | 6.00                   | 6.02               | 0.02      | 0.0620                 |
| B+C: | 164 | 2.50                   | 2.53               | 0.03      | 0.0261                 |
| B-C: | 164 | 1.50                   | 1.52               | 0.02      | 0.0216                 |

s.d.(AB) S(between runs): 0.049

Sw(within run): 0.044

S/Sw: 1.1

s.d.(BC) S(between runs): 0.017

Sw(within run): 0.015

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |       |     |     |
|------|---|-------|-----|-----|
| 9.66 | - | 10.34 | for | A+B |
| 5.75 | - | 6.25  | for | A-B |
| 2.38 | - | 2.62  | for | B+C |
| 1.41 | - | 1.59  | for | B-C |

## DUPLICATES:

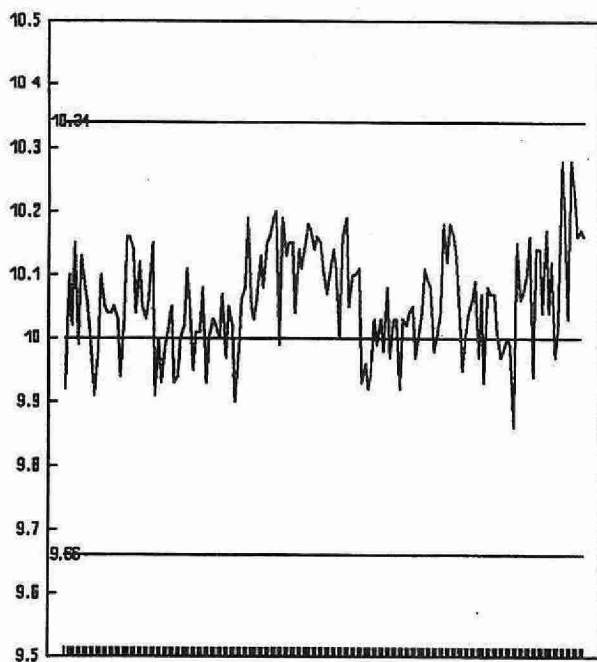
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 33           | 0.00 - 1.00               | 0.0096                 | 2.0                         |
| 26           | 1.01 - 2.00               | 0.0118                 | 1.2                         |
| 143          | 2.01 - 5.00               | 0.0372                 | 1.8                         |
| 165          | 5.01 - 10.0               | 0.0700                 | 0.8                         |
| 367          | Overall                   | 0.0471                 |                             |

## OTHER CHECKS:

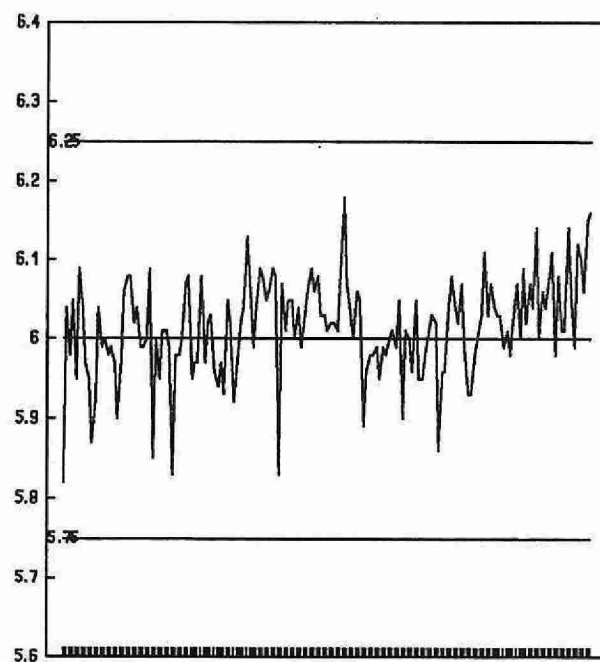
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 164 | 0.0002 | 0.0025                 |

MAGNESIUM (mg/L as Mg)

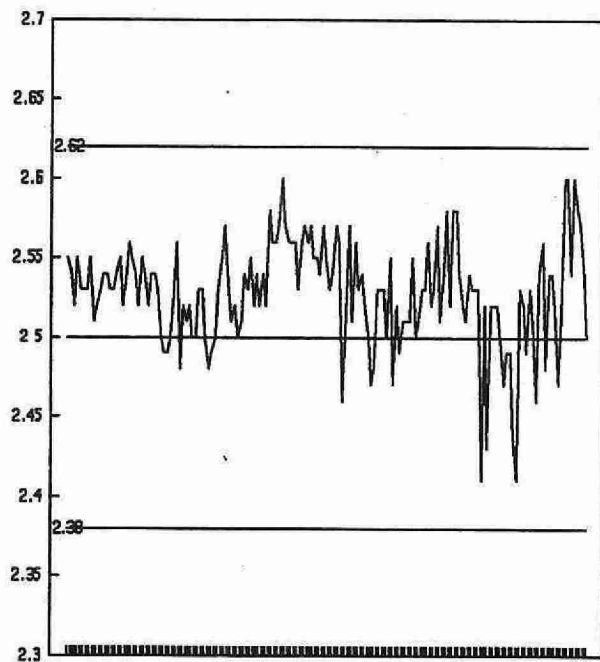
QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94



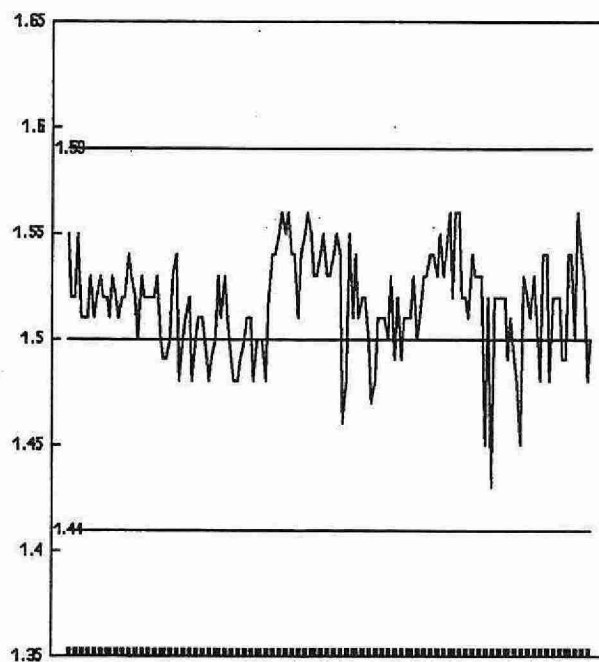
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## MAGNESIUM

### IDENTIFICATION:

|                      |  |                   |            |
|----------------------|--|-------------------|------------|
| Laboratory Unit      | Atomic Absorption  | Method Introduced | 08/04/86   |
| Method Reference No. | E3217A   | Units             | mg/L as Mg |
| LIMS Product Code    | CAT3217,CATS3217,HARD3217  | Supervisor        | J. McBride |
| Sample Type/Matrix   | Domestic Waters, Leachates, Effluents, Sewage, Industrial Wastes |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.87 at the full scale level.

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 11 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA                   |
| Drift       | BL every 10 samples; 2 standards every 20 samples. |

# MAGNESIUM

QUALITY CONTROL DATA FROM 04/01/94 TO 29/12/94

Laboratory Unit: Absorption

Full Scale: to 50.00 mg/L as Mg

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 115 | 40.0                   | 40.004             | -0.004    | 0.5679                 |
| B:   | 115 | 10.0                   | 10.069             | -0.069    | 0.1889                 |
| C:   | 115 | 2.5                    | 2.503              | -0.003    | 0.0742                 |
| A+B: | 115 | 50.0                   | 50.074             | -0.074    | 0.6522                 |
| A-B: | 115 | 30.0                   | 29.935             | 0.065     | 0.5393                 |
| B+C: | 115 | 12.5                   | 12.572             | -0.072    | 0.2286                 |
| B-C: | 115 | 7.5                    | 7.566              | -0.066    | 0.1735                 |

s.d.(AB) S(between runs): 0.42

Sw(within run): 0.38

S/Sw: 1.1

s.d.(BC) S(between runs): 0.14

Sw(within run): 0.12

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 47.80 | - | 52.20 | for | A+B |
| 28.50 | - | 31.50 | for | A-B |
| 11.45 | - | 13.55 | for | B+C |
| 6.80  | - | 8.20  | for | B-C |

## DUPLICATES:

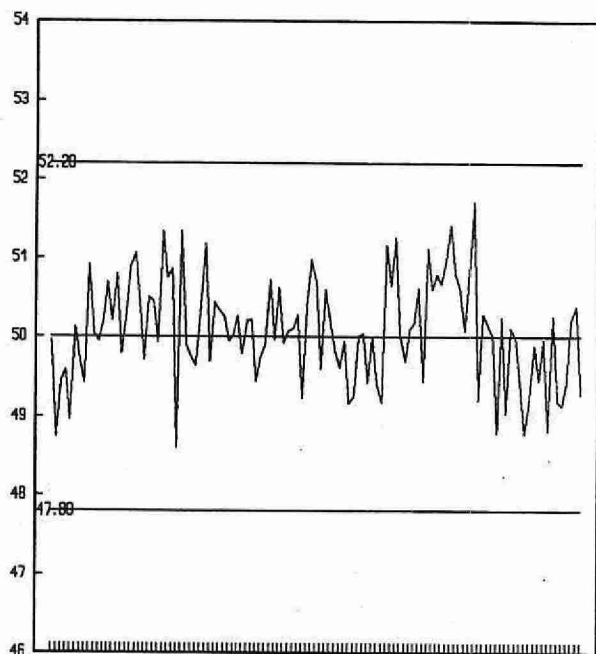
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 59           | 0.00 - 5.00               | 0.0648                 | 2.6                         |
| 58           | 5.01 - 10.00              | 0.1284                 | 1.4                         |
| 100          | 10.01 - 25.00             | 0.2959                 | 2.4                         |
| 74           | 25.01 - 50.00             | 0.4708                 | 2.9                         |
| 291          | Overall                   | 0.2405                 |                             |

## OTHER CHECKS:

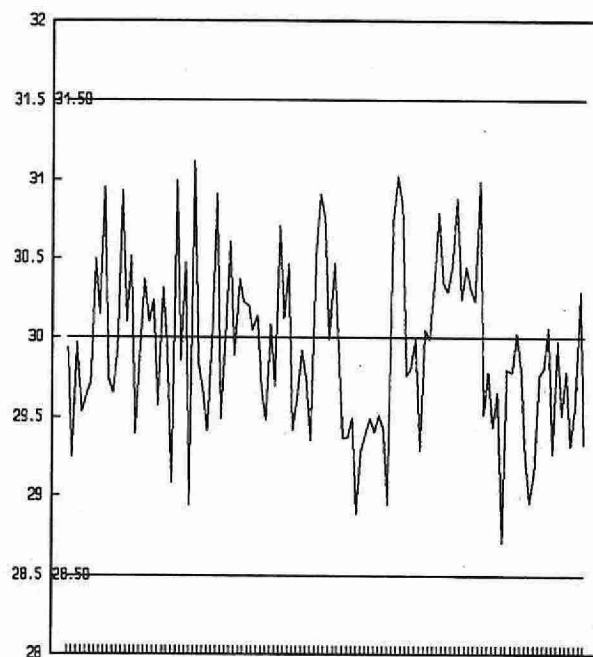
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 115 | -0.118 | 0.1259                 |

# MAGNESIUM (mg/L as Mg)

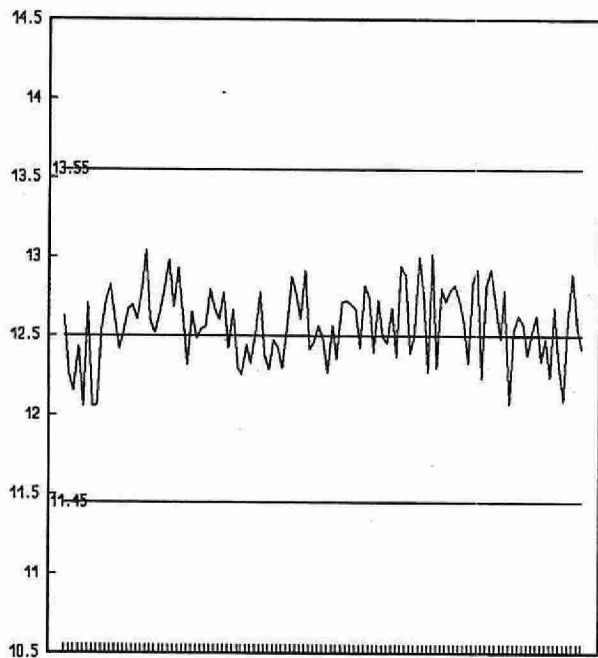
QUALITY CONTROL DATA FROM 04/01/94 TO 29/12/94



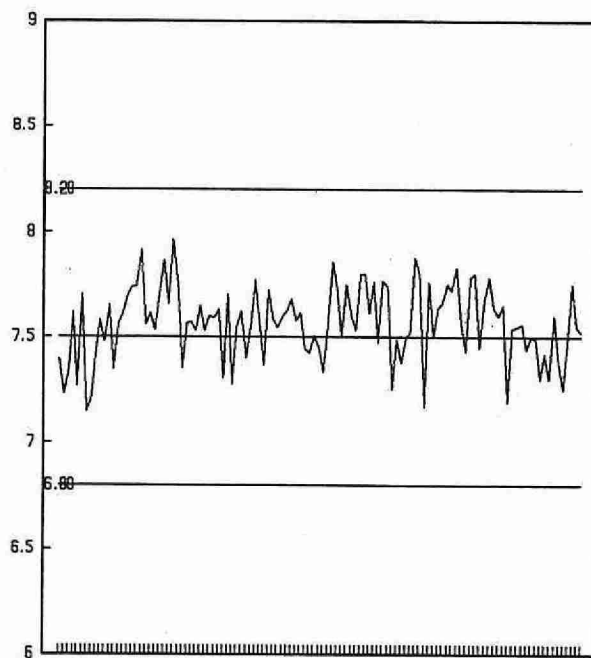
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## MAGNESIUM

### IDENTIFICATION:

|                      |                |                   |            |
|----------------------|----------------|-------------------|------------|
| Laboratory Unit      | Dorset         | Method Introduced | 20/07/88   |
| LIS Test Name Code   | MGUR           | Units             | mg/L as Mg |
| Work Station Code    | DOFLAME        | Unit Code         | 064812     |
| Method Code          | 001CA1         | Supervisor        | J. McBride |
| Method Reference No. | E3249A         |                   |            |
| Sample Type/Matrix   | Rivers, Lakes, |                   |            |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 5 mL    |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.005 | Current T value: 0.025 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA       |
| Drift       | BL, reslope standard every 10 samples. |

### NOTES:

The control standards are corrected for the LTB from which they were made.

# MAGNESIUM

QUALITY CONTROL DATA FROM 17/01/94 TO 22/12/94

Laboratory Unit: Dorset

Full Scale: to 2.0 mg/L as Mg

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 74 | 1.6                    | 1.607              | 0.007     | 0.0128                 |
| B:   | 74 | 0.4                    | 0.401              | 0.001     | 0.0046                 |
| C:   | 74 | 0.1                    | 0.105              | 0.005     | 0.0026                 |
| A+B: | 74 | 2.0                    | 2.008              | 0.008     | 0.0141                 |
| A-B: | 74 | 1.2                    | 1.206              | 0.006     | 0.0131                 |
| B+C: | 74 | 0.5                    | 0.505              | 0.005     | 0.0055                 |
| B-C: | 74 | 0.3                    | 0.296              | -0.004    | 0.0049                 |

s.d.(AB) S(between runs): 0.010

Sw(within run): 0.009

S/Sw: 1.0

s.d.(BC) S(between runs): 0.004

Sw(within run): 0.003

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 1.96  | - | 2.04  | for | A+B |
| 1.17  | - | 1.23  | for | A-B |
| 0.483 | - | 0.517 | for | B+C |
| 0.313 | - | 0.287 | for | B-C |

## DUPLICATES:

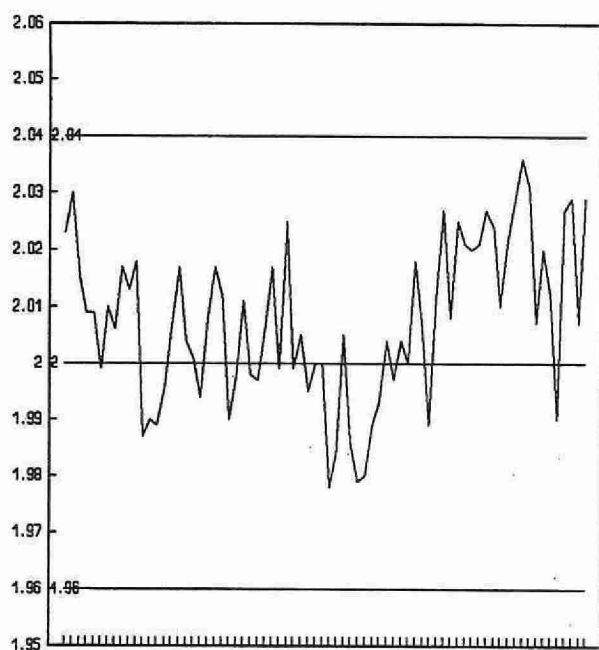
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 58           | 0.00 - 0.20               | 0.0017                 | 7.9                         |
| 19           | 0.21 - 0.40               | 0.0094                 | 3.8                         |
| 119          | 0.41 - 1.00               | 0.0156                 | 3.9                         |
| 21           | 1.01 - 2.00               | 0.0304                 | 3.4                         |
| 217          | Overall                   | 0.0110                 |                             |

## OTHER CHECKS:

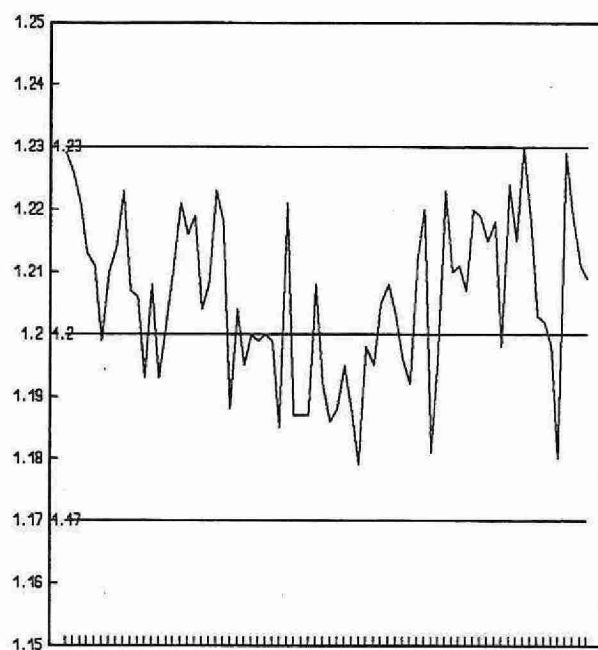
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 74 | 0.0006 | 0.0013                 |

# MAGNESIUM (mg/L as Mg)

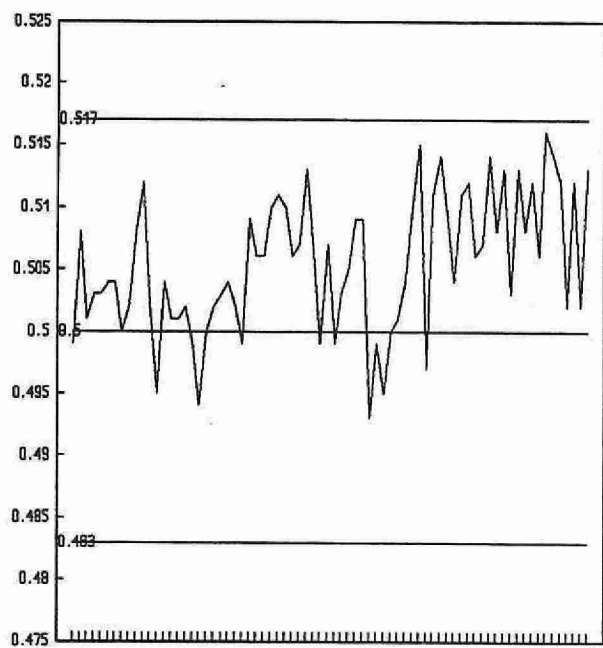
QUALITY CONTROL DATA FROM 17/01/94 TO 22/12/94



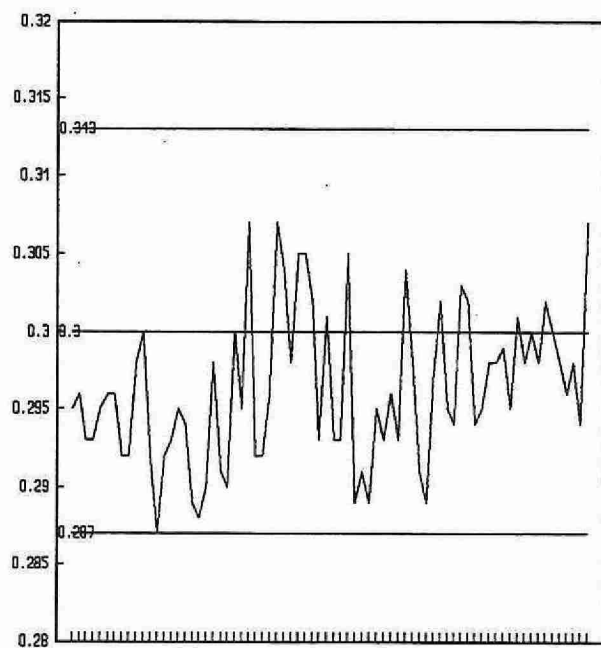
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT



## MANGANESE, TOTAL

### IDENTIFICATION:

|                      |  |                   |            |
|----------------------|--|-------------------|------------|
| Laboratory Unit      | Dorset                                       | Method Introduced | 1991       |
| LIS Test Name Code   | MNUT   | Units             | µg/L as Mn |
| Work Station Code    | DOFEMN                                       | Unit Code         | 063825     |
| Method Code          | 504BC2                                       | Supervisor        | J. McBride |
| Method Reference No. | E3303B                                       |                   |            |
| Sample Type/Matrix   | Surface water, precipitation, soil leachates |                   |            |

### SAMPLING:

|                   |  |
|-------------------|--|
| Quantity Required | 25 mL  |
| Container         | Glass or plastic, capped, acidified to 0.25% with HNO <sub>3</sub> |

### ANALYTICAL PROCEDURE:

An undigested sample is introduced to an in-line UV digester. A reducing agent and an ammonium buffer are added to the sample. Formaldoxime complexes with Mn to develop a colour the intensity of which is proportional to the concentration of Mn in the sample. EDTA is then added to complex interferences. The color is read at 480nm. A reference channel is used to counter the effects of residual natural colour in the sample. In the reference channel the EDTA is added prior to the addition of colour reagent.

### INSTRUMENTATION:

- An AAII autoanalyzer with colorimeter and automated sampler.

### REPORTING:

|                                |                    |                    |
|--------------------------------|--------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 1 | Current T value: 5 |
|--------------------------------|--------------------|--------------------|

### CALIBRATION:

BL plus 4 standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | Long Term blank, 3 QC's, 4 duplicates   |
| Drift       | Blank plus 1 standard every 10 samples. |

# MANGANESE, TOTAL

QUALITY CONTROL DATA FROM 10/01/94 TO 20/12/94

Laboratory Unit: Dorset

Full Scale: to 200.0 µg/L as Mn

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 39 | 150.0                  | 149.75             | -0.25     | 1.262                  |
| B:   | 39 | 50.0                   | 50.13              | 0.13      | 1.389                  |
| C:   | 39 | 10.0                   | 10.37              | 0.37      | 0.553                  |
| A+B: | 39 | 200.0                  | 199.93             | -0.07     | 2.126                  |
| A-B: | 39 | 100.0                  | 99.62              | -0.38     | 1.686                  |
| B+C: | 39 | 60.0                   | 60.60              | 0.60      | 1.625                  |
| B-C: | 39 | 40.0                   | 39.81              | -0.19     | 1.483                  |

s.d.(AB) S(between runs): 1.33

Sw(within run): 1.19

S/Sw: 1.1

s.d.(BC) S(between runs): 1.10

Sw(within run): 1.05

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|        |   |        |     |     |
|--------|---|--------|-----|-----|
| 193.27 | - | 206.73 | for | A+B |
| 94.95  | - | 105.05 | for | A-B |
| 55.16  | - | 64.84  | for | B+C |
| 36.37  | - | 43.63  | for | B-C |

## DUPLICATES:

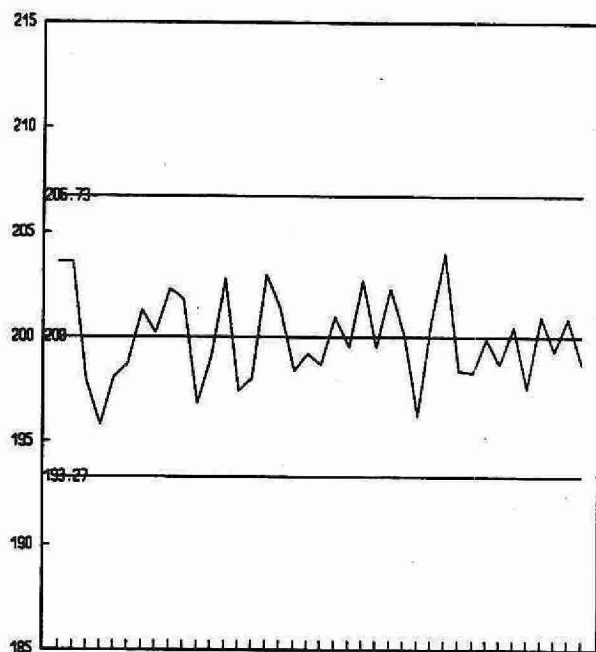
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 61           | 0.0 - 20.0                | 0.6959                 | 8.8                         |
| 46           | 20.1 - 40.0               | 0.9827                 | 3.2                         |
| 39           | 40.1 - 100.0              | 1.2606                 | 1.8                         |
| 9            | 100.1 - 200.0             | 3.2897                 | 1.9                         |
| 155          | Overall                   | 1.0256                 |                             |

## OTHER CHECKS:

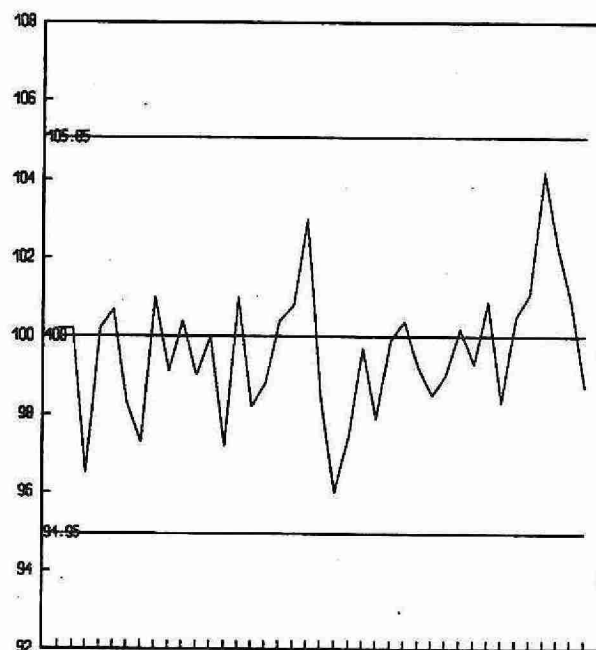
|                 | n  | Mean    | Standard Deviation (1) |
|-----------------|----|---------|------------------------|
| Long Term Blank | 39 | -0.0256 | 0.2673                 |

# MANGANESE, TOTAL ( $\mu\text{g/L}$ as Mn)

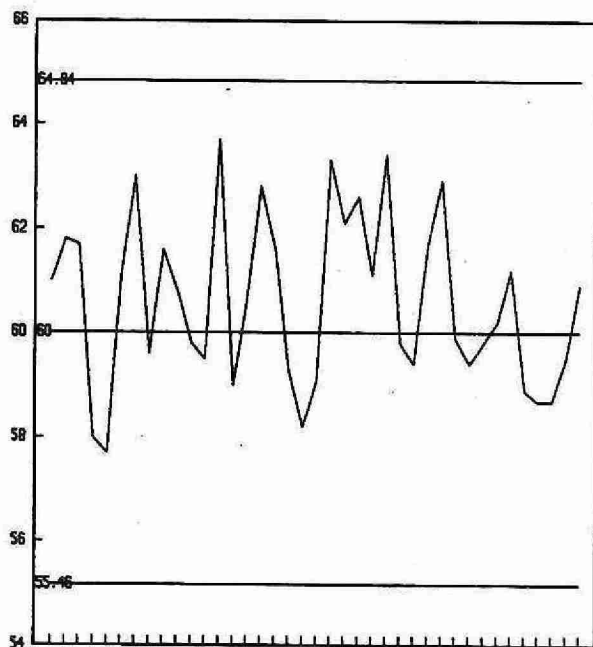
QUALITY CONTROL DATA FROM 10/01/94 TO 20/12/94



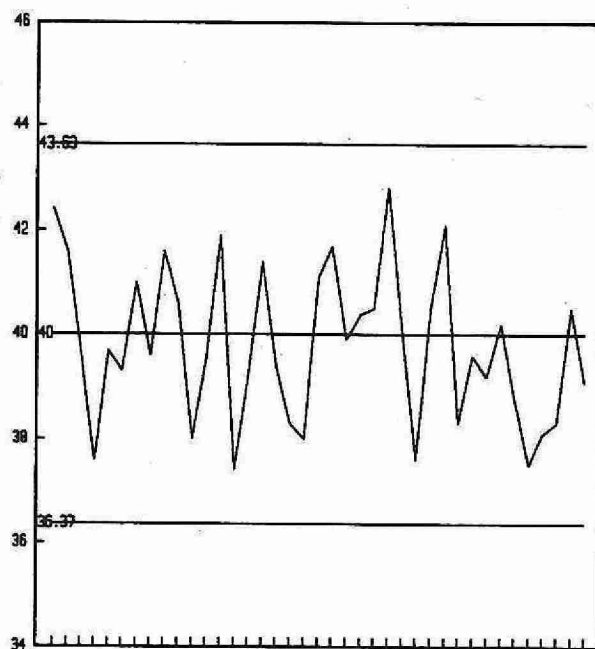
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## NITROGEN, AMMONIA PLUS AMMONIUM

### IDENTIFICATION:

|                     |                                      |                   |             |
|---------------------|--------------------------------------|-------------------|-------------|
| Laboratory Unit     | Colourimetry                         | Method Introduced | 01/05/84    |
| Method Reference No | E3149A                               | Units             | mg/L as N   |
| LIMS Product Code   | AMM3149                              | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Precipitation, Throughfall, Stemflow |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects. Ammonia plus ammonium for dry deposition air filter extracts is also determined using this method.

Approximate absorbance: 0.7 at the full scale level.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5cm light path at 630 nm. Data capture, reduction and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                        |                       |
|--------------------------------|------------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.002 | Current T value: 0.01 |
|--------------------------------|------------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g. QCA                |
| Drift       | BL every 10 samples; standard every 20 samples |

### NOTES:

Filter Extracts: Ammonia plus ammonium ions are determined on an extract from a dry deposition air filter. The analytical values are multiplied by 25 and reported as µg/filter as N. QC data outlined for this method may be applied to these results by multiplying the QC data by 25.

# NITROGEN, AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 12/01/94 TO 15/12/94

Laboratory Unit: Colourimetry

Full Scale: to 2.00 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 46 | 1.600                  | 1.597              | -0.003    | 0.0125                 |
| B:   | 46 | 0.800                  | 0.804              | 0.004     | 0.0082                 |
| C:   | 46 | 0.160                  | 0.161              | 0.001     | 0.0061                 |
| A+B: | 46 | 2.400                  | 2.401              | 0.001     | 0.0159                 |
| A-B: | 46 | 0.800                  | 0.794              | -0.006    | 0.0140                 |
| B+C: | 46 | 0.960                  | 0.965              | 0.005     | 0.0116                 |
| B-C: | 46 | 0.640                  | 0.642              | 0.002     | 0.0087                 |

s.d.(AB) S(between runs): 0.011

Sw(within run): 0.010

S/Sw: 1.1

s.d.(BC) S(between runs): 0.007

Sw(within run): 0.006

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 2.31  | - | 2.49  | for | A+B |
| 0.74  | - | 0.86  | for | A-B |
| 0.92  | - | 1.00  | for | B+C |
| 0.616 | - | 0.664 | for | B-C |

## DUPLICATES:

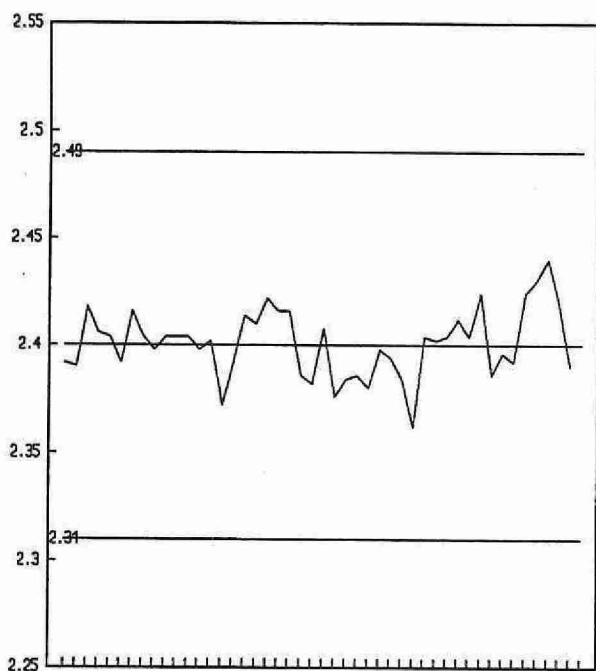
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 56           | 0.000 - 0.200             | 0.0023                 | 6.1                         |
| 28           | 0.201 - 0.400             | 0.0047                 | 1.4                         |
| 34           | 0.401 - 1.000             | 0.0046                 | 0.9                         |
| 15           | 1.001 - 2.000             | 0.0010                 | 0.6                         |
| 133          | Overall                   | 0.0043                 |                             |

## OTHER CHECKS:

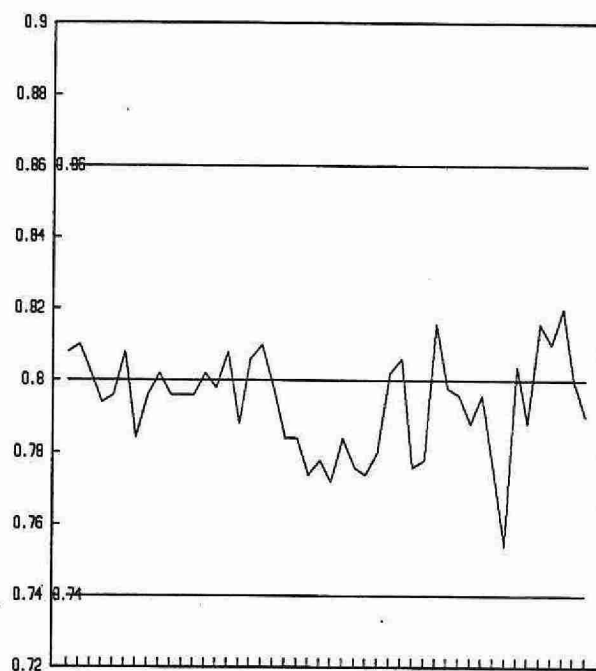
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 41 | 0.000 | 0.0052                 |

# NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N)

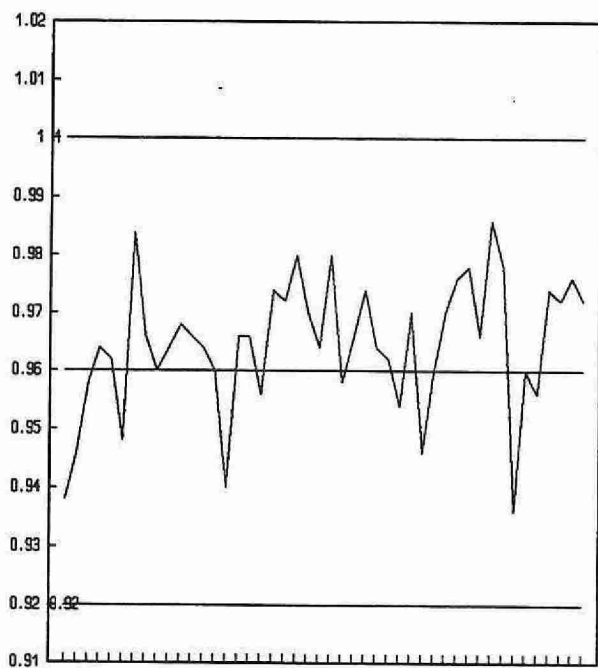
QUALITY CONTROL DATA FROM 12/01/94 TO 15/12/94



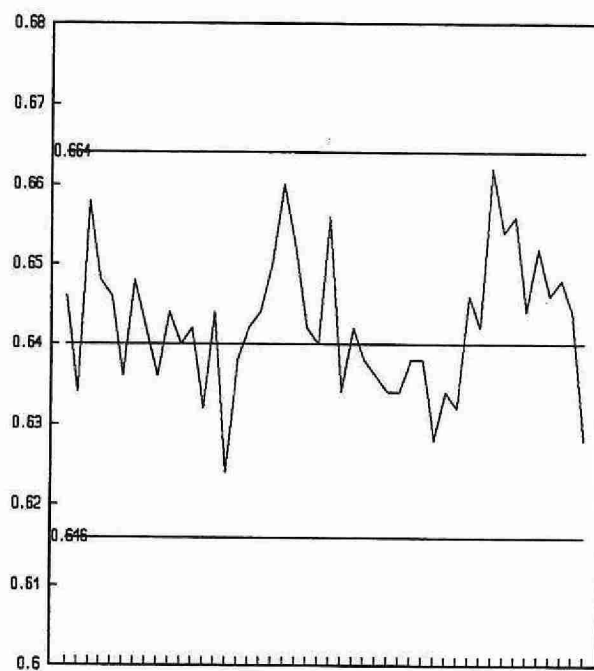
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## NITROGEN, AMMONIA PLUS AMMONIUM

### IDENTIFICATION:

|                     |   |                   |             |
|---------------------|---|-------------------|-------------|
| Laboratory Unit     | Colourimetry                            | Method Introduced | 01/04/78    |
| Method Reference No | E3364A                                  | Units             | mg/L as N   |
| LIMS Product Code   | DISNUT3364                              | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Rivers, Lakes, Soil Extracts, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance: 0.5 at the full scale level.

Nitrate plus nitrite, nitrite, and reactive orthophosphate are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                        |                       |
|--------------------------------|------------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.002 | Current T value: 0.01 |
|--------------------------------|------------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g. QCA                |
| Drift       | BL every 10 samples; standard every 20 samples |

### NOTES:

Sept.'94 the method codes RNDNP-E3174A, E3175A, E3208A and E3266 were amalgamated and a new method code RNDNP-E3364A was generated.



# NITROGEN, AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 07/01/94 TO 05/12/94

Laboratory Unit: Colourimetry

Full Scale: to 2.00 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 94 | 1.60                      | 1.5995                | -0.0005   | 0.0125                    |
| B:   | 94 | 0.800                     | 0.797                 | -0.003    | 0.0075                    |
| C:   | 94 | 0.160                     | 0.163                 | 0.003     | 0.0058                    |
| A+B: | 94 | 2.40                      | 2.396                 | -0.004    | 0.0154                    |
| A-B: | 94 | 0.800                     | 0.803                 | 0.003     | 0.0137                    |
| B+C: | 94 | 0.960                     | 0.9601                | 0.0001    | 0.0104                    |
| B-C: | 94 | 0.640                     | 0.634                 | -0.006    | 0.0084                    |

s.d.(AB) S(between runs): 0.0103

Sw(within run): 0.0097

S/Sw: 1.1

s.d.(BC) S(between runs): 0.0067

Sw(within run): 0.0060

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 2.31  | - | 2.49  | for | A+B |
| 0.740 | - | 0.860 | for | A-B |
| 0.920 | - | 1.00  | for | B+C |
| 0.616 | - | 0.664 | for | B-C |

## DUPLICATES:

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 250             | 0.000 - 0.200                | 0.0085                    | 36.6                           |
| 13              | 0.201 - 0.400                | 0.0209                    | 18.8                           |
| 12              | 0.401 - 1.00                 | 0.0549                    | 9.4                            |
| 0               | 1.01 - 2.00                  | N.A.                      | N.A.                           |
| 275             | Overall                      | 0.0095                    |                                |

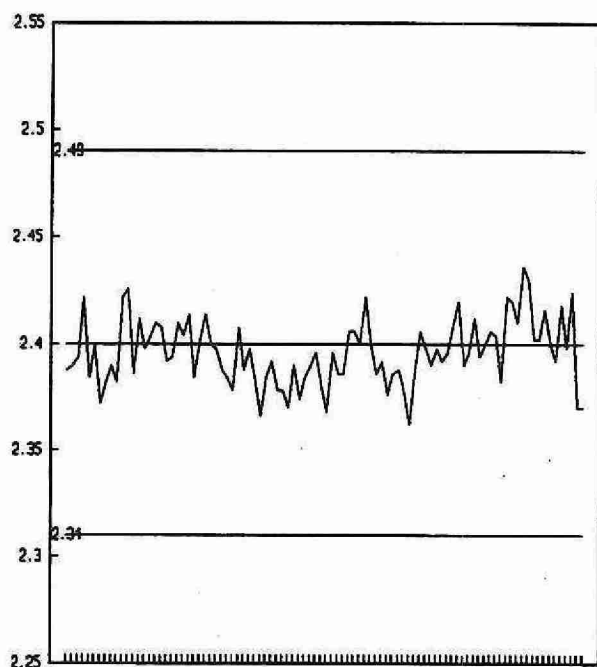
## OTHER CHECKS:

|                 | n  | Mean   | Standard<br>Deviation (1) |
|-----------------|----|--------|---------------------------|
| Long Term Blank | 94 | 0.0002 | 0.0057                    |

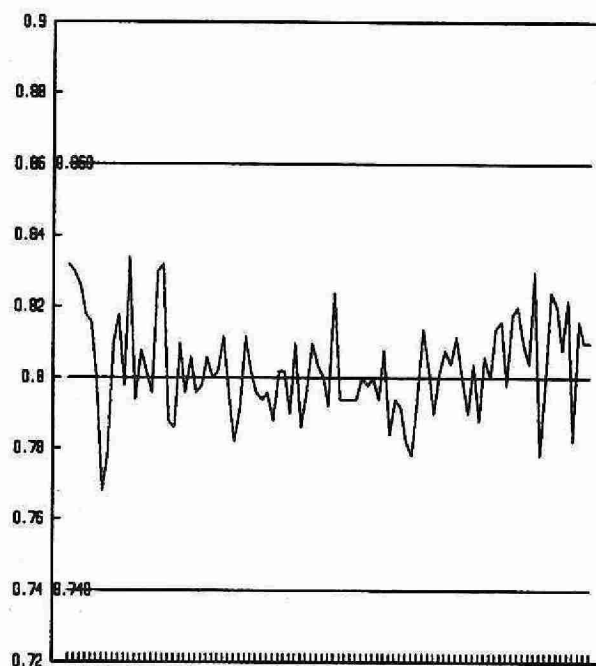


# NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N)

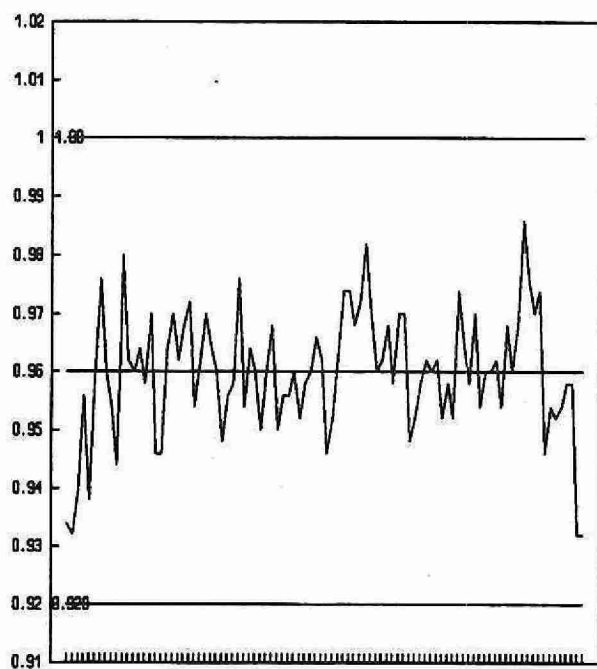
QUALITY CONTROL DATA FROM 07/01/94 TO 05/12/94



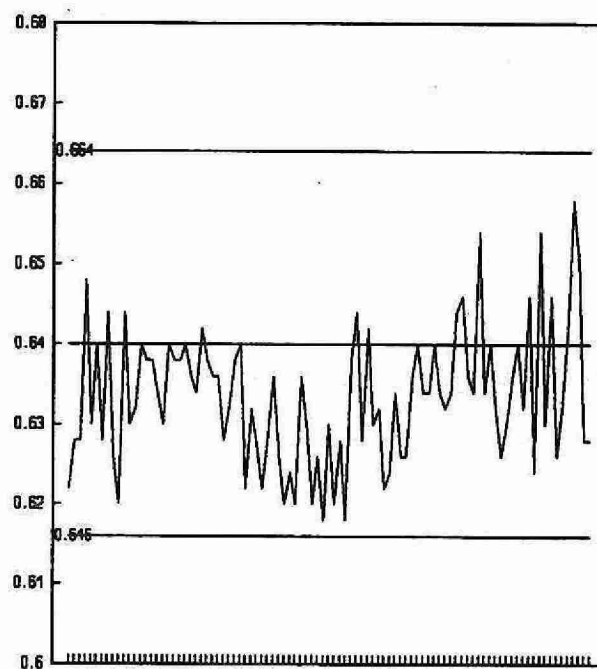
QUALITY CONTROL SAMPLE A+B



QUALITY CONTROL SAMPLE A-B



QUALITY CONTROL SAMPLE B+C



QUALITY CONTROL SAMPLE B-C

CONTROL LIMIT

## NITROGEN, AMMONIA PLUS AMMONIUM

### IDENTIFICATION:

|                     |  |                   |             |
|---------------------|--|-------------------|-------------|
| Laboratory Unit     | Colourimetry   | Method Introduced | 01/04/77    |
| Method Reference No | E3366A   | Units             | mg/L as N   |
| LIMS Product Code   | DISNUT3366   | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Sewage, Industrial Waste, Leachate, Domestic Waters, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.7 at the full scale level.

Reactive orthophosphate, nitrogen-nitrite and nitrogen-nitrate plus nitrite are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus one 38°C heating bath (7.7 mL delay).

Colourimetric measurement is through a 1.5 cm. light path at 630 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g. QCA                |
| Drift       | BL every 10 samples; standard every 20 samples |

### NOTES:

Sept.'94 the method codes SDNP-E3223A, E3193A, E3184A and E3185 were amalgamated and a new method code SDNP-E3366A was generated.

# NITROGEN, AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 07/01/94 TO 20/12/94

Laboratory Unit: Colourimetry

Full Scale: to 50.0 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 82 | 40.0                   | 40.04              | 0.04      | 0.2306                 |
| B:   | 82 | 20.0                   | 20.03              | 0.03      | 0.1288                 |
| C:   | 82 | 4.00                   | 3.995              | -0.005    | 0.0449                 |
| A+B: | 82 | 60.0                   | 60.07              | 0.07      | 0.2879                 |
| A-B: | 82 | 20.0                   | 20.001             | 0.001     | 0.2380                 |
| B+C: | 82 | 24.0                   | 24.03              | 0.03      | 0.1497                 |
| B-C: | 82 | 16.0                   | 16.04              | 0.04      | 0.1217                 |

s.d.(AB) S(between runs): 0.19  
s.d.(BC) S(between runs): 0.097

Sw(within run): 0.17  
Sw(within run): 0.086

S/Sw: 1.1  
S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 58.8 | - | 61.2 | for | A+B |
| 19.1 | - | 20.9 | for | A-B |
| 23.3 | - | 24.7 | for | B+C |
| 15.5 | - | 16.5 | for | B-C |

## DUPLICATES:

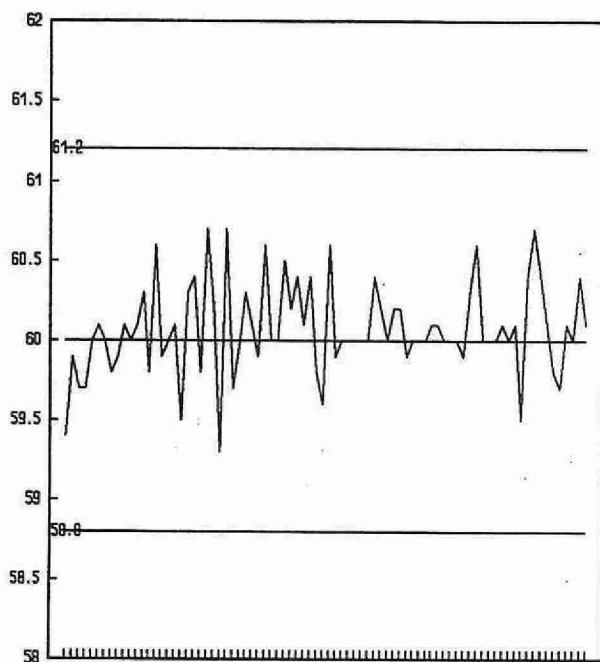
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 171          | 0.00 - 5.00               | 0.0615                 | 13.5                        |
| 16           | 5.10 - 10.0               | 0.1439                 | 2.2                         |
| 47           | 10.1 - 25.0               | 0.3517                 | 3.1                         |
| 3            | 25.1 - 50.0               | 0.0816                 | N.A.                        |
| 237          | Overall                   | 0.0684                 |                             |

## OTHER CHECKS:

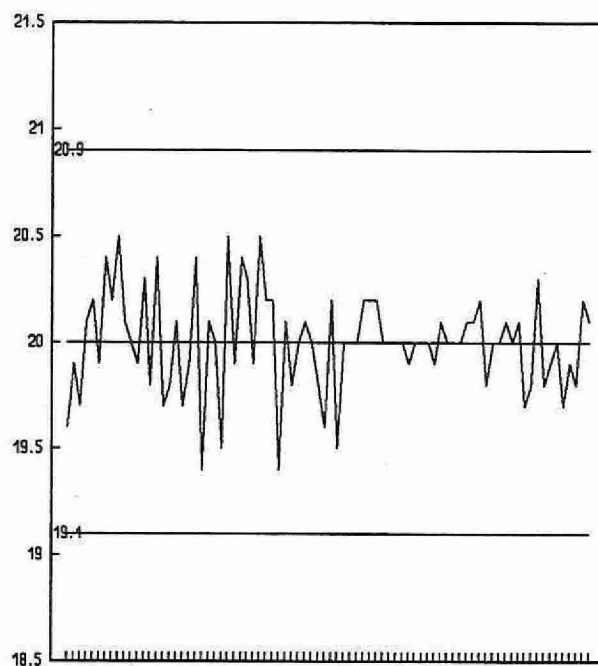
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 82 | 0.0049 | 0.0365                 |

# NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N)

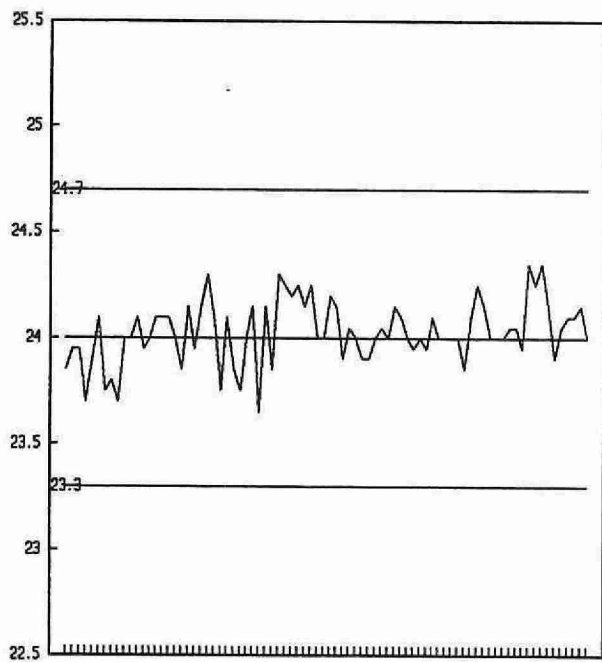
QUALITY CONTROL DATA FROM 07/01/94 TO 20/12/94



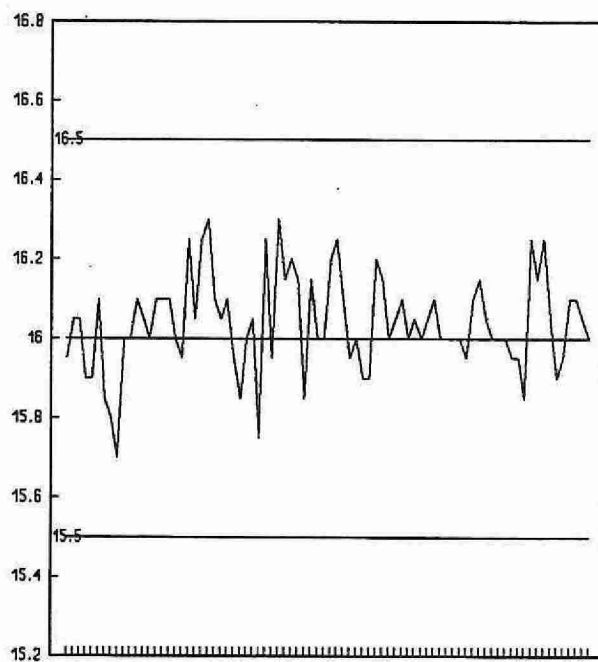
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## NITROGEN, AMMONIA PLUS AMMONIUM

### IDENTIFICATION:

|                      |   |                   |            |
|----------------------|---|-------------------|------------|
| Laboratory Unit      | Dorset  | Method Introduced | 01/06/76   |
| LIS Test Name Code   | NNHTFR  | Units             | µg/L as N  |
| Work Station Code    | DONUT   | Unit Code         | 063807     |
| Method Code          | 1524C2  | Supervisor        | J. McBride |
| Method Reference No. | E3374A  |                   |            |
| Sample Type/Matrix:  | Streams, Lakes, Precipitation, and Soil Leachates |                   |            |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: | 50 mL            |
| Container:         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance : 0.40 at the full scale level.

Nitrate plus nitrite is determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37°C heating bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Two analytical ranges are obtained from the output of the colourimeter.

### REPORTING:

|                                |                    |                    |
|--------------------------------|--------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 1 | Current T value: 5 |
|--------------------------------|--------------------|--------------------|

### CALIBRATION:

BL plus 8 standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 QC standards, e.g. QCA                              |
| Drift       | BL every 10 samples and BL plus check standard every 20 samples |

### NOTES:

JAN. 1995 LIMS replaced LIS and the method reference no. was changed from E3033A to E3374A. LIMS product code is AMMNO3374.

# NITROGEN, AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 13/01/94 TO 22/12/94

Laboratory: Dorset

Full Scale: to 1000 µg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 57 | 600                    | 600.32             | 0.32      | 5.1449                 |
| B:   | 57 | 200                    | 200.35             | 0.35      | 3.5732                 |
| C:   | 57 | 60                     | 59.60              | -0.40     | 2.9692                 |
| A+B: | 57 | 800                    | 800.67             | 0.67      | 7.7927                 |
| A-B: | 57 | 400                    | 399.97             | -0.03     | 4.2129                 |
| B+C: | 57 | 260                    | 259.95             | -0.05     | 5.5627                 |
| B-C: | 57 | 140                    | 140.75             | 0.75      | 3.4963                 |

s.d.(AB) S(between runs): 4.43

Sw(within run): 2.98

S/Sw: 1.5

s.d.(BC) S(between runs): 3.28

Sw(within run): 2.47

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

|     |   |     |     |     |
|-----|---|-----|-----|-----|
| 780 | - | 820 | for | A+B |
| 380 | - | 420 | for | A-B |
| 245 | - | 275 | for | B+C |
| 125 | - | 155 | for | B-C |

## DUPLICATES:

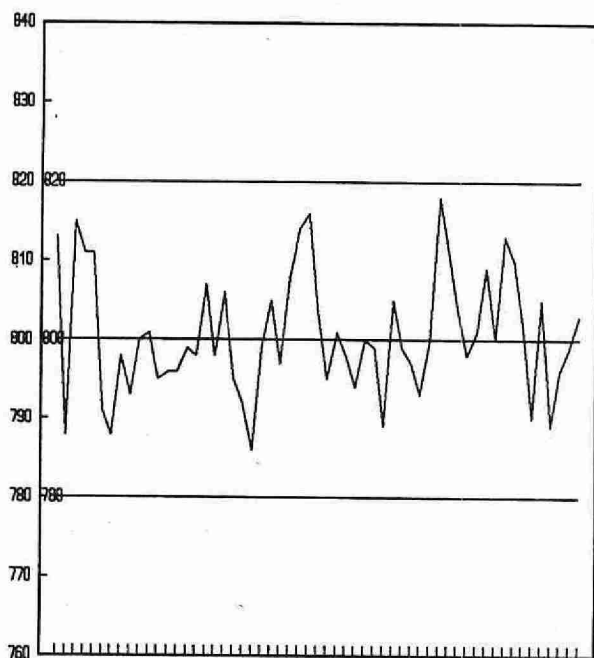
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 148          | 0.0 - 100                 | 1.8576                 | 6.9119                      |
| 12           | 101 - 200                 | 2.2910                 | 1.3928                      |
| 3            | 201 - 500                 | 3.3665                 | 1.2183                      |
| 1            | 501 - 1000                | N.A.                   | N.A.                        |
| 164          | Overall                   | 1.9037                 |                             |

## OTHER CHECKS:

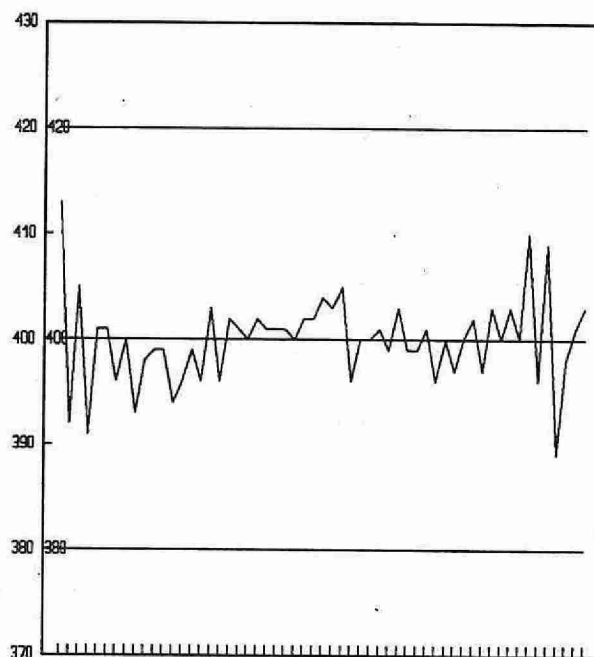
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 57 | 0.526 | 0.6841                 |

# NITROGEN, AMMONIA PLUS AMMONIUM ( $\mu\text{g/L as N}$ )

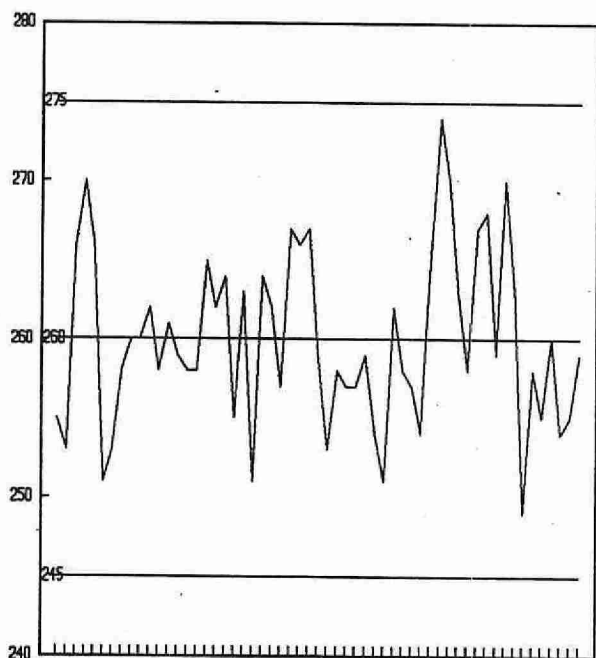
QUALITY CONTROL DATA FROM 13/01/94 TO 22/12/94



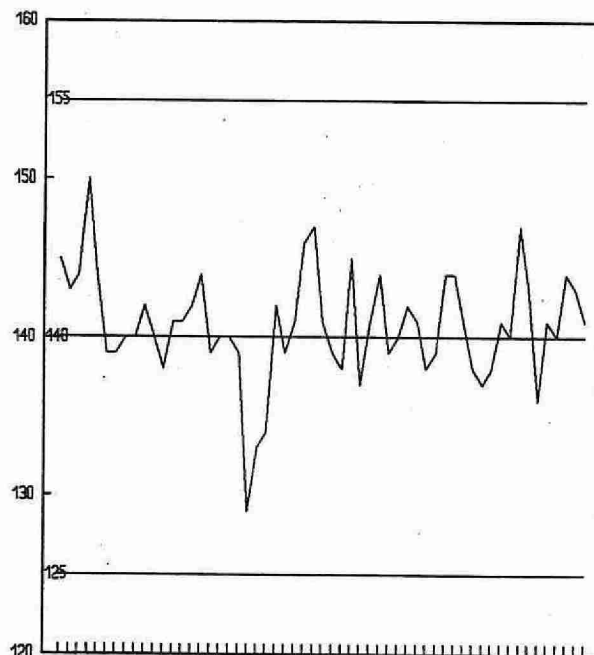
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT



## NITROGEN, NITRATE

### IDENTIFICATION:

|                     |  |                   |                |
|---------------------|--|-------------------|----------------|
| Laboratory Unit     | Ion Chromatography   | Method Introduced | 01/07/80       |
| Method Reference No | E3148A   | Units             | µg/Filter as N |
| LIMS Product Code   | LOV3148, ANLOV3148,<br>NYL3148, TEF3148, ANION3148   | Supervisor        | F. Lo          |
| Sample Type/Matrix  | W40 filters from LoVol filter packs, Teflon and Nylon filters from LoVol and Sequential filter packs |                   |                |

### SAMPLING:

|                   |                          |
|-------------------|--------------------------|
| Quantity Required | 1 filter                 |
| Container         | 50 mL polypropylene tube |

### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW (W40) or 25.0 mL of DDW (Teflon) or 25.0 mL of 0.03N NaOH (Nylon) in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with  $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$  to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by the comparison of the sample peak heights to a series of standards. Results are converted to µg/filter as N. Chloride and sulphate are determined simultaneously.

### INSTRUMENTATION:

Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

### REPORTING:

|                                |                            |                            |
|--------------------------------|----------------------------|----------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.01 mg/L | Current T value: 0.05 mg/L |
|--------------------------------|----------------------------|----------------------------|

### CALIBRATION:

BL plus 9 standards

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift       | 1 standard every 10 samples     |

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received. To convert unit from mg/L to µg/Filter, the concentration of N in mg/L is multiplied by 50 for W40 filters or 25 for Teflon and Nylon filters.



# NITROGEN, NITRATE

QUALITY CONTROL DATA FROM 19/01/94 TO 06/12/94

Laboratory Unit: Ion Chromatography

Full Scale: to 2.0 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 40 | 1.60                      | 1.612                 | 0.012     | 0.0188                    |
| B:   | 40 | 0.40                      | 0.397                 | -0.003    | 0.0055                    |
| A+B: | 40 | 2.00                      | 2.010                 | 0.010     | 0.0209                    |
| A-B: | 40 | 1.20                      | 1.215                 | 0.015     | 0.0182                    |

s.d.(AB)    S(between runs): 0.0138    Sw(within run): 0.0130    S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

1.93 - 2.07 for A+B  
1.15 - 1.25 for A-B

## DUPLICATES:

For W40 filters:

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 12              | 0.00 - 0.20                  | 0.0024                    | 2.4                            |
| 8               | 0.21 - 0.40                  | 0.0034                    | 1.3                            |
| 17              | 0.41 - 1.20                  | 0.0039                    | 0.5                            |
| 2               | 1.21 - 2.00                  | 0.0026                    | 0.2                            |
| 39              | Overall                      | 0.0032                    |                                |

For Teflon filters:

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 45              | 0.00 - 0.20                  | 0.0027                    | 8.4                            |
| 7               | 0.21 - 0.40                  | 0.0028                    | 0.8                            |
| 9               | 0.41 - 1.20                  | 0.0032                    | 0.5                            |
| 3               | 1.21 - 2.00                  | 0.0027                    | 0.2                            |
| 64              | Overall                      | 0.0029                    |                                |

# NITROGEN, NITRATE

QUALITY CONTROL DATA FROM 19/01/94 TO 06/12/94

Laboratory Unit: Ion Chromatography

Full Scale: to 2.0 mg/L as N

## DUPLICATES:

For Nylon filters:

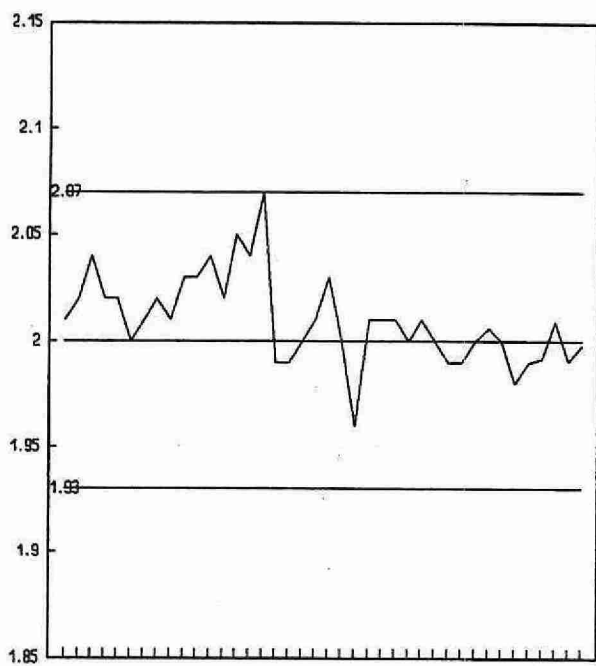
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 35              | 0.00 - 0.20                  | 0.0051                    | 8.1                            |
| 26              | 0.21 - 0.40                  | 0.0047                    | 1.5                            |
| 18              | 0.41 - 1.20                  | 0.0064                    | 0.9                            |
| 0               | 1.21 - 2.00                  | N.A.                      | N.A.                           |
| 79              | Overall                      | 0.0053                    |                                |

## OTHER CHECKS:

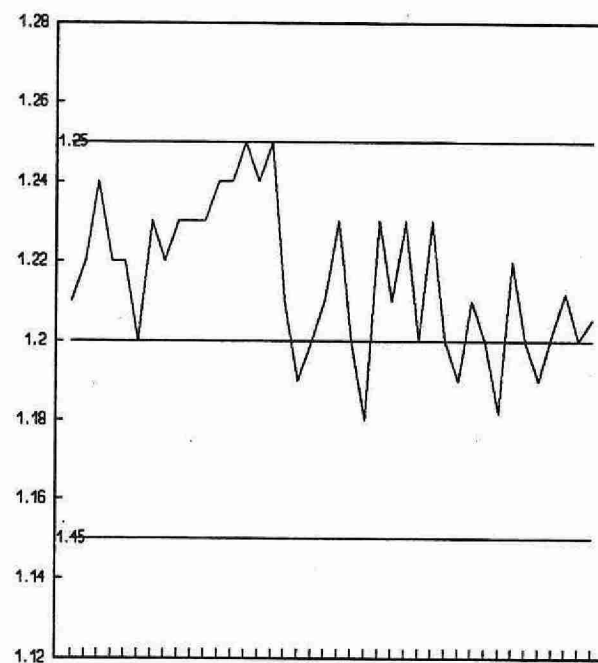
|                 | n  | Mean   | Standard<br>Deviation (1) |
|-----------------|----|--------|---------------------------|
| Long Term Blank | 40 | 0.0005 | 0.0029                    |

NITROGEN, NITRATE (mg/L as N)

QUALITY CONTROL DATA FROM 19/01/94 TO 06/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## NITROGEN, NITRATE

### IDENTIFICATION:

|                     |                                      |                   |           |
|---------------------|--------------------------------------|-------------------|-----------|
| Laboratory Unit     | Ion Chromatography                   | Method Introduced | 01/04/78  |
| Method Reference No | E3372A                               | Units             | mg/L as N |
| LIMS Product Code   | ANION3372                            | Supervisor        | F. Lo     |
| Sample Type/Matrix  | Precipitation, Throughfall, Stemflow |                   |           |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 15 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with  $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$  to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by the comparison of the sample peak heights to a series of standards.

Sulphate and chloride are determined simultaneously.

### INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.01 | Current T value: 0.05 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift       | 1 standard every 10 samples     |

### NOTES:

Formerly method E3147A, method number changed for LIMS in 1993 to E3372A.

# NITROGEN, NITRATE

QUALITY CONTROL DATA FROM 11/01/94 TO 16/12/94

Laboratory Unit: Ion Chromatography

Full Scale: to 1.0 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 35 | 0.80                      | 0.801                 | 0.001     | 0.0060                    |
| B:   | 35 | 0.20                      | 0.193                 | -0.007    | 0.0102                    |
| A+B: | 35 | 1.00                      | 0.994                 | -0.006    | 0.0147                    |
| A-B: | 35 | 0.60                      | 0.607                 | 0.007     | 0.0081                    |

s.d.(AB)      S(between runs): 0.0084      Sw(within run): 0.0057      S/Sw: 1.5

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.96 - 1.04 for A+B  
0.57 - 0.63 for A-B

## DUPLICATES:

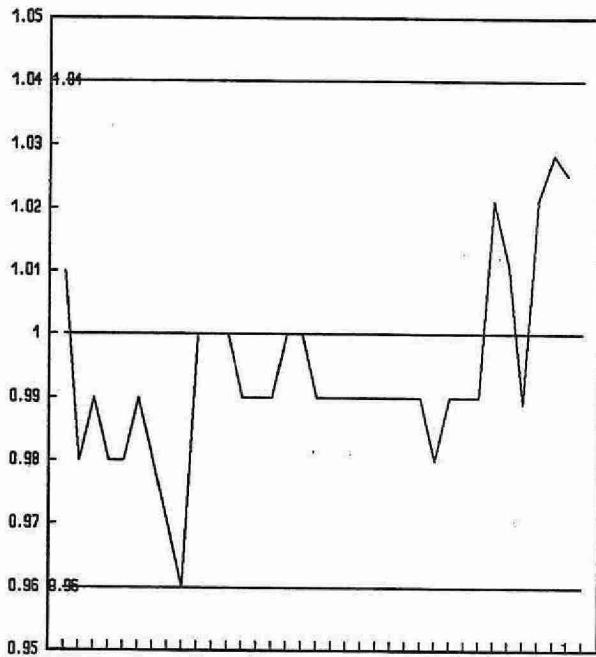
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 4               | 0.00 - 0.20                  | 0.0058                    | 7.6                            |
| 14              | 0.21 - 0.50                  | 0.0045                    | 2.1                            |
| 14              | 0.51 - 1.00                  | 0.0087                    | 2.8                            |
| 32              | Overall                      | 0.0062                    |                                |

## OTHER CHECKS:

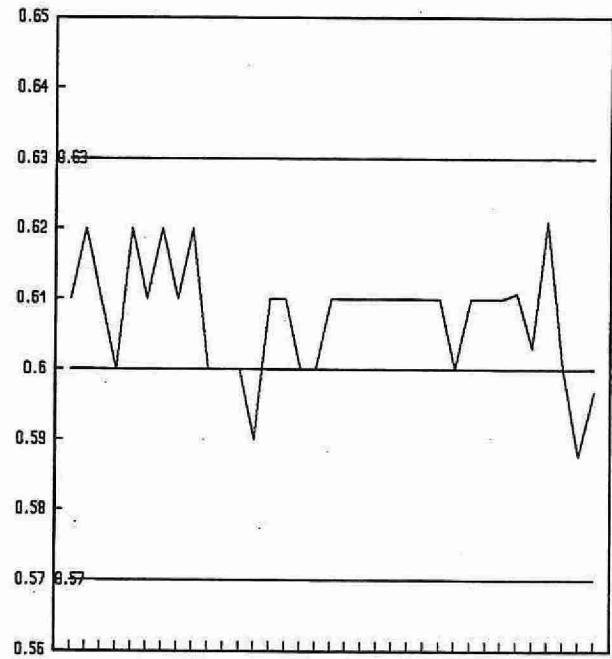
|                 | n  | Mean  | Standard<br>Deviation (1) |
|-----------------|----|-------|---------------------------|
| Long Term Blank | 35 | 0.000 | 0.0000                    |

NITROGEN, NITRATE (mg/L as N)

QUALITY CONTROL DATA FROM 11/01/94 TO 16/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## NITROGEN, NITRATE PLUS NITRITE

### IDENTIFICATION:

|                     |  |                   |             |
|---------------------|--|-------------------|-------------|
| Laboratory Unit     | Colourimetry   | Method Introduced | 01/04/78    |
| Method Reference No | E3364A   | Units             | mg/L as N   |
| LIMS Product Code   | DISNUT3364   | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Rivers, Lakes, Precipitation, Soil Extracts, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance: 0.6 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive orthophosphate are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm. light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.005 | Current T value: 0.025 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|              |  |
|--------------|--|
| Calibration  | LTBL plus 3 standards, e.g. QCA  |
| Drift        | BL every 10 samples; standard every 20 samples   |
| Interference | Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression. |
| Recovery     | Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.              |

### NOTES:

Sept.'94 the method codes RNDNP-E3174A, E3175A, E3208A and E3266 were amalgamated and a new method code RNDNP-E3364A was generated.

# NITROGEN, NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 07/01/94 TO 04/12/94

Laboratory Unit: Colourimetry

Full Scale: to 5.00 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 94 | 4.00                   | 3.995              | -0.005    | 0.0326                 |
| B:   | 94 | 2.00                   | 2.005              | 0.005     | 0.0201                 |
| C:   | 94 | 0.400                  | 0.406              | 0.006     | 0.0092                 |
| A+B: | 94 | 6.00                   | 6.00               | 0.00      | 0.0432                 |
| A-B: | 94 | 2.00                   | 1.99               | -0.01     | 0.0326                 |
| B+C: | 94 | 2.40                   | 2.41               | -0.01     | 0.0257                 |
| B-C: | 94 | 1.60                   | 1.5995             | -0.0005   | 0.0177                 |

s.d.(AB) S(between runs): 0.027

Sw(within run): 0.023

S/Sw: 1.2

s.d.(BC) S(between runs): 0.016

Sw(within run): 0.013

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 5.77 | - | 6.23 | for | A+B |
| 1.85 | - | 2.15 | for | A-B |
| 2.30 | - | 2.50 | for | B+C |
| 1.54 | - | 1.66 | for | B-C |

## DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 188          | 0.000 - 0.500             | 0.0072                 | 11.7                        |
| 31           | 0.501 - 1.00              | 0.0150                 | 6.7                         |
| 34           | 1.01 - 2.50               | 0.0311                 | 7.1                         |
| 15           | 2.51 - 5.00               | 0.0534                 | 2.4                         |
| 268          | Overall                   | 0.0126                 |                             |

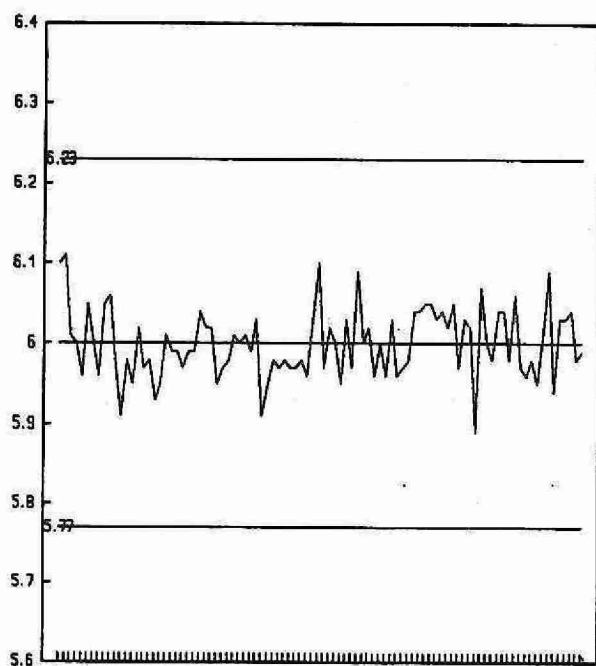
## OTHER CHECKS:

|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 94 | 0.0003 | 0.0064                 |

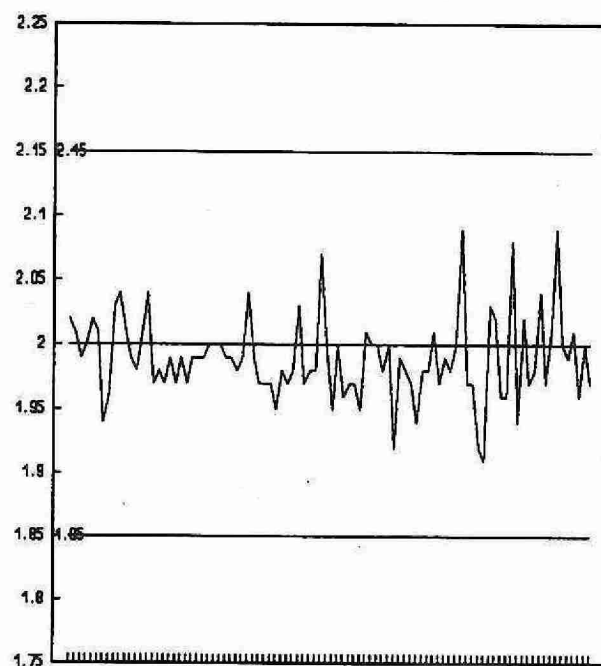


# NITROGEN, NITRATE PLUS NITRITE (mg/L as N)

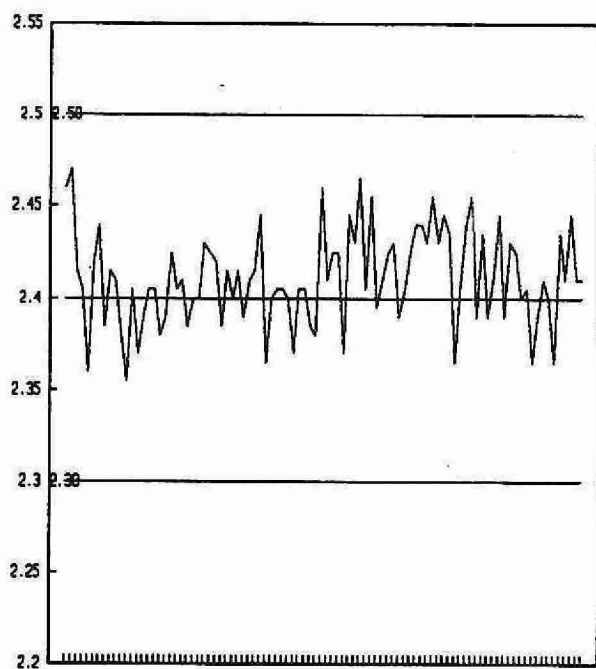
QUALITY CONTROL DATA FROM 07/01/94 TO 04/12/94



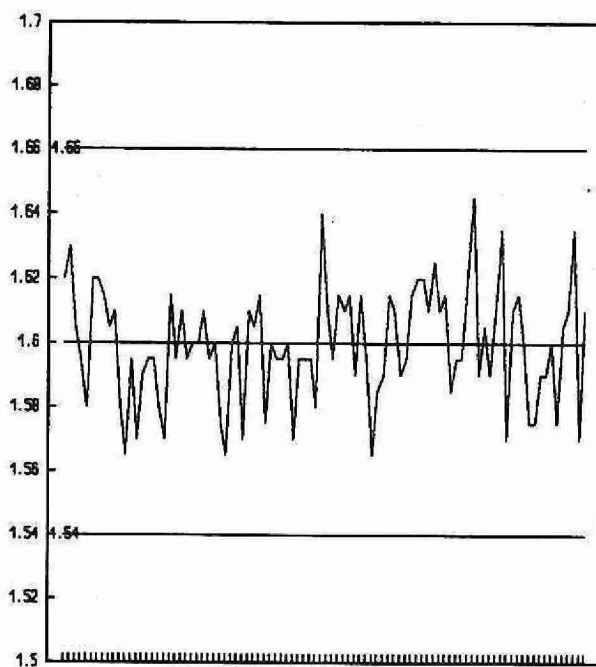
QUALITY CONTROL SAMPLE A+B



QUALITY CONTROL SAMPLE A-B



QUALITY CONTROL SAMPLE B+C



QUALITY CONTROL SAMPLE B-C

CONTROL LIMIT

## NITROGEN, NITRATE PLUS NITRITE

### IDENTIFICATION:

|                     |   |                   |             |
|---------------------|---|-------------------|-------------|
| Laboratory Unit     | Colourimetry  | Method Introduced | 01/04/78    |
| Method Reference No | E3366A  | Units             | mg/L as N   |
| LIMS Product Code   | DISNUT3366  | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Sewage, Industrial Waste, Leachate, Domestic Waters |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 38°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance: 0.7 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive phosphate are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 38°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Two analytical ranges are obtained from the output of the colourimeter. Data capture, reduction, and processing via a multi - stage microcomputer system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|              |  |
|--------------|--|
| Calibration  | LTBL plus 3 standards, e.g. QCA  |
| Drift        | BL every 10 samples; standard every 20 samples   |
| Interference | Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression. |
| Recovery     | Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.              |

### NOTES:

Sept.'94 the method codes SDNP-E3223A, E3193A, E3184A and E3185 were amalgamated and a new method code SDNP-E3366A was generated.

# NITROGEN, NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 05/01/94 TO 20/12/94

Laboratory Unit: Colourimetry

Full Scale: to 50.0 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 82 | 40.0                   | 40.04              | 0.04      | 0.2586                 |
| B:   | 82 | 20.0                   | 20.01              | 0.01      | 0.1771                 |
| C:   | 82 | 4.00                   | 4.002              | 0.002     | 0.0643                 |
| A+B: | 82 | 60.0                   | 60.05              | 0.05      | 0.3594                 |
| A-B: | 82 | 20.0                   | 20.03              | 0.03      | 0.2594                 |
| B+C: | 82 | 24.0                   | 24.01              | 0.01      | 0.2105                 |
| B-C: | 82 | 16.0                   | 16.04              | 0.04      | 0.1633                 |

s.d.(AB) S(between runs): 0.22

Sw(within run): 0.18

S/Sw: 1.2

s.d.(BC) S(between runs): 0.13

Sw(within run): 0.12

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 58.7 | - | 61.3 | for | A+B |
| 19.0 | - | 21.0 | for | A-B |
| 23.3 | - | 24.7 | for | B+C |
| 15.5 | - | 16.5 | for | B-C |

## DUPLICATES:

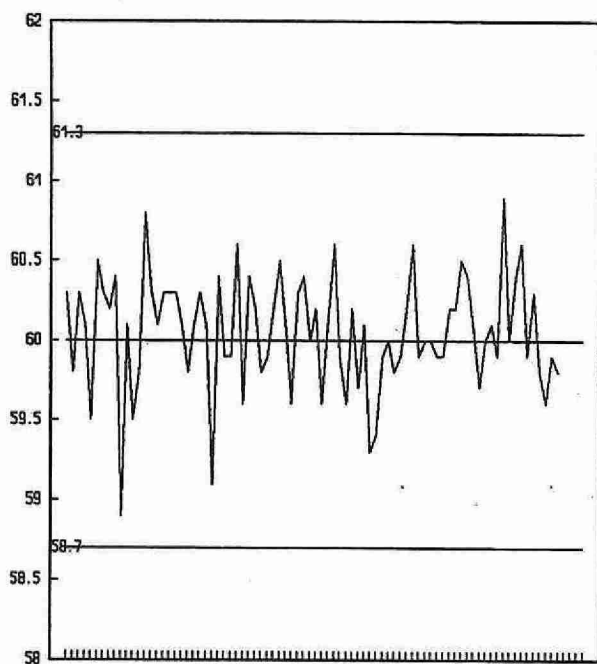
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 187          | 0.000 - 5.00              | 0.0436                 | 11.3                        |
| 17           | 5.01 - 10.0               | 0.1112                 | 1.3                         |
| 33           | 10.1 - 25.0               | 0.2476                 | 1.6                         |
| 8            | 25.1 - 50.0               | 0.1856                 | 0.5                         |
| 245          | Overall                   | 0.1225                 |                             |

## OTHER CHECKS:

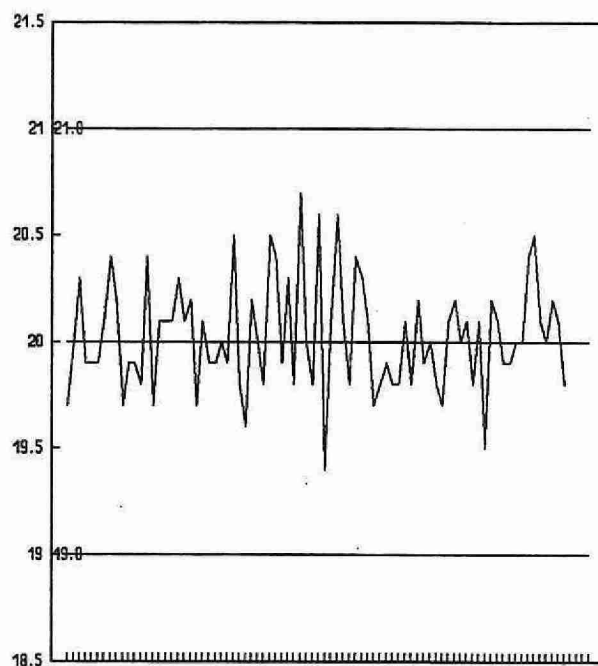
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 82 | 0.0073 | 0.0409                 |

# NITROGEN, NITRATE PLUS NITRITE (mg/L as N)

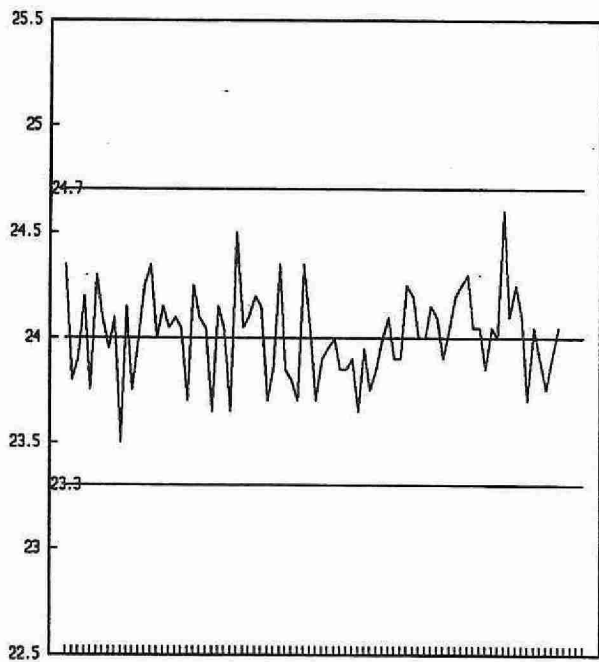
QUALITY CONTROL DATA FROM 05/01/94 TO 20/12/94



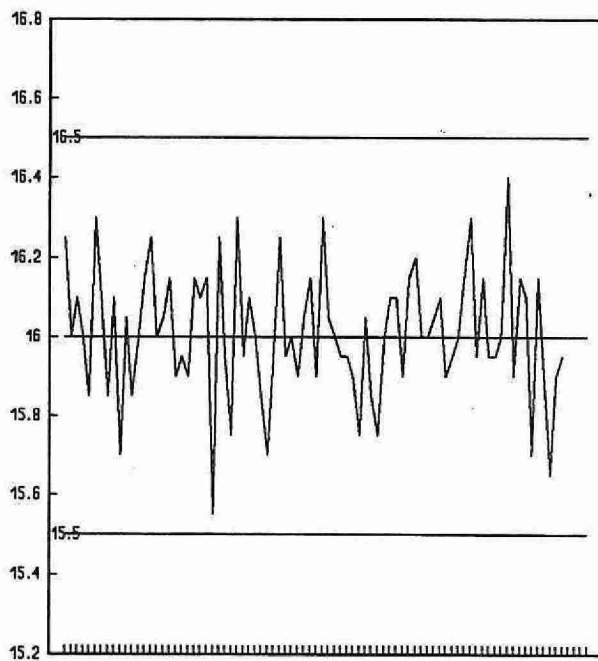
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## NITROGEN, NITRATE PLUS NITRITE

### IDENTIFICATION:

|                     |                                  |                   |             |
|---------------------|----------------------------------|-------------------|-------------|
| Laboratory Unit     | Colourimetry                     | Method Introduced | 01/04/76    |
| Method Reference No | E3369A                           | Units             | mg/L as N   |
| LIMS Product Code   | FNOT3369                         | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Ministry of Health Water Samples |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 38°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance: 0.5 at the full scale level.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.1 | Current T value: 0.5 |
|--------------------------------|----------------------|----------------------|

### CALIBRATION:

BL plus 6 standards

### CONTROLS:

|              |  |
|--------------|--|
| Calibration  | LTB plus 3 standards, e.g. QCA   |
| Drift        | BL every 10 samples; standard every 20 samples   |
| Interference | Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression. |
| Recovery     | Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.              |

### NOTES:

Sept.'94 the method codes WFNO3-E3221A, and E3220A were amalgamated and a new method code WFNO3-E3369A was generated.

# NITROGEN, NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 05/01/94 TO 22/12/94

Laboratory Unit: Colourimetry

Analytical Range: to 20.0 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 75 | 16.0                   | 16.004             | -0.004    | 0.1309                 |
| B:   | 75 | 8.0                    | 8.036              | -0.036    | 0.0747                 |
| C:   | 75 | 1.60                   | 1.597              | 0.003     | 0.0434                 |
| A+B: | 75 | 24.0                   | 24.040             | -0.040    | 0.1516                 |
| A-B: | 75 | 8.0                    | 7.968              | 0.032     | 0.1499                 |
| B+C: | 75 | 9.60                   | 9.633              | -0.033    | 0.0860                 |
| B-C: | 75 | 6.40                   | 6.439              | -0.039    | 0.0868                 |

s.d.(AB)    S(between runs): 0.107                      Sw(within run): 0.106                      S/Sw: 1.0

s.d.(BC)    S(between runs): 0.061                      Sw(within run): 0.061                      S/Sw: 1.0

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 23.2 | - | 24.8 | for | A+B |
| 7.4  | - | 8.6  | for | A-B |
| 9.2  | - | 10.0 | for | B+C |
| 6.0  | - | 6.8  | for | B-C |

## DUPLICATES:

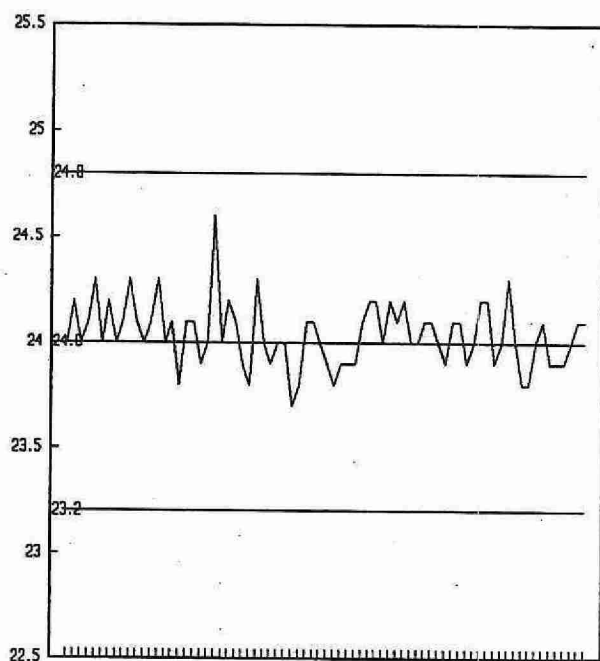
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 150             | 0.0 - 2.0                    | 0.0497                    | 12.7                           |
| 22              | 2.1 - 4.0                    | 0.0452                    | 1.5                            |
| 38              | 4.1 - 10.0                   | 0.1736                    | 2.6                            |
| 9               | 10.1 - 20.0                  | 0.1563                    | 1.1                            |
| 219             | Overall                      | 0.0902                    |                                |

## OTHER CHECKS:

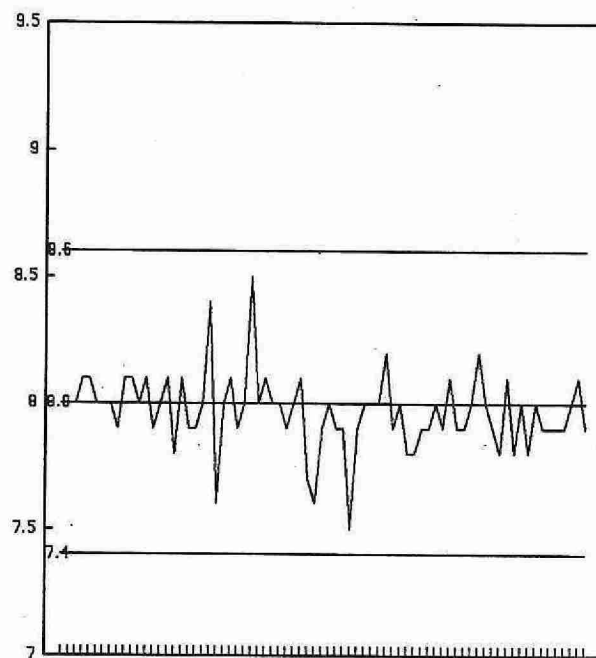
|                 | n  | Data<br>Mean | Standard (1)<br>Deviation |
|-----------------|----|--------------|---------------------------|
| Long Term Blank | 75 | 0.0027       | 0.0231                    |

# NITROGEN, NITRATE PLUS NITRITE (mg/L as N)

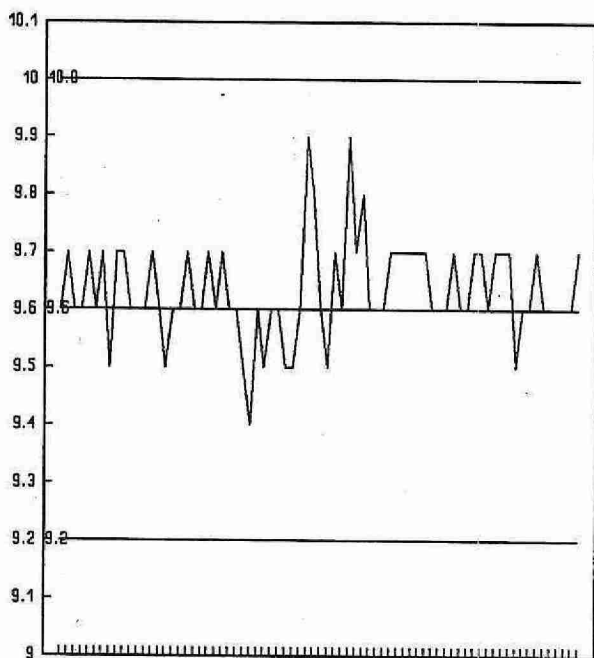
QUALITY CONTROL DATA FROM 05/01/94 TO 22/12/94



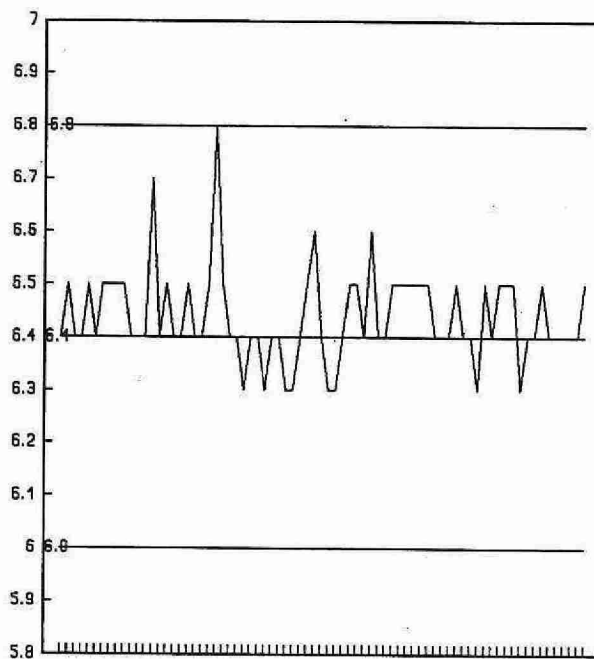
QUALITY CONTROL SAMPLE A+B



QUALITY CONTROL SAMPLE A-B



QUALITY CONTROL SAMPLE B+C



QUALITY CONTROL SAMPLE B-C

CONTROL LIMIT



## NITROGEN, NITRATE PLUS NITRITE

### IDENTIFICATION:

|                      |   |                   |            |
|----------------------|---|-------------------|------------|
| Laboratory Unit      | Dorset  | Method Introduced | 13/06/78   |
| LIS Test Name Code   | NNOTFR  | Units             | µg/L as N  |
| Work Station Code    | DONUT   | Unit Code         | 063807     |
| Method Code          | 1525C2  | Supervisor        | J. McBride |
| Method Reference No. | E3374A  |                   |            |
| Sample Type/Matrix:  | Streams, Lakes, Precipitation, and Soil Leachates |                   |            |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: | 50 mL            |
| Container:         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a sample. Nitrate is reduced to nitrite in alkaline media at 37°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance : 0.4 at the full scale level.  
Ammonia plus ammonium is determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 5.0 cm. light path at 520 nm.

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |
|--------------------------------|--------------------|---------------------|

### CALIBRATION:

BL plus 8 standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 QC standards, e.g. QCA                              |
| Drift       | BL every 10 samples and BL plus check standard every 20 samples |

### NOTES:

JAN. 1995 LIMS replaced LIS and the method reference no. was changed from E3034A to E3374A. LIMS product code is AMMNO3374.



# NITROGEN, NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 13/01/94 TO 21/12/94

Laboratory: Dorset

Full Scale: to 1000 µg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 57 | 600                    | 602.49             | 2.49      | 5.8221                 |
| B:   | 57 | 200                    | 200.37             | 0.37      | 3.0801                 |
| C:   | 57 | 60                     | 59.32              | -0.68     | 2.5083                 |
| A+B: | 57 | 800                    | 802.86             | 2.86      | 7.2369                 |
| A-B: | 57 | 400                    | 402.12             | 2.12      | 5.8648                 |
| B+C: | 57 | 260                    | 259.68             | -0.32     | 4.6834                 |
| B-C: | 57 | 140                    | 141.05             | 1.05      | 3.1020                 |

s.d.(AB) S(between runs): 4.66

Sw(within run): 4.15

S/Sw: 1.1

s.d.(BC) S(between runs): 2.81

Sw(within run): 2.19

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

|     |   |     |         |
|-----|---|-----|---------|
| 780 | - | 820 | for A+B |
| 380 | - | 420 | for A-B |
| 245 | - | 275 | for B+C |
| 125 | - | 155 | for B-C |

## DUPLICATES:

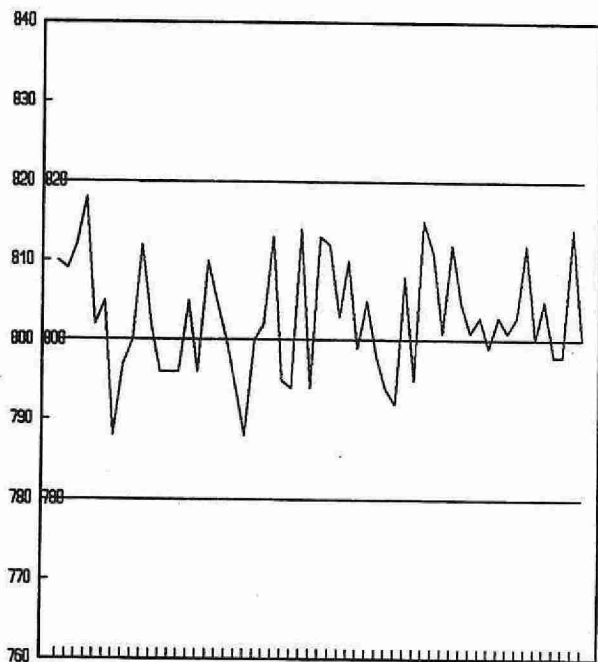
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 97           | 0.0 - 100                 | 2.0487                 | 6.3                         |
| 38           | 101 - 200                 | 3.6793                 | 2.4                         |
| 21           | 201 - 500                 | 4.5866                 | 1.6                         |
| 3            | 501 - 1000                | 2.4495                 | 0.3                         |
| 159          | Overall                   | 2.8972                 |                             |

## OTHER CHECKS:

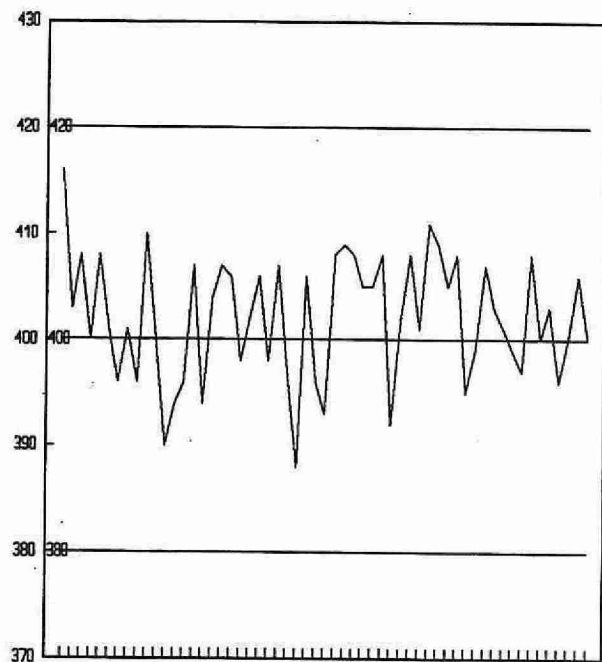
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 57 | 0.035 | 0.1840                 |

# NITROGEN, NITRATE PLUS NITRITE ( $\mu\text{g/L}$ as N)

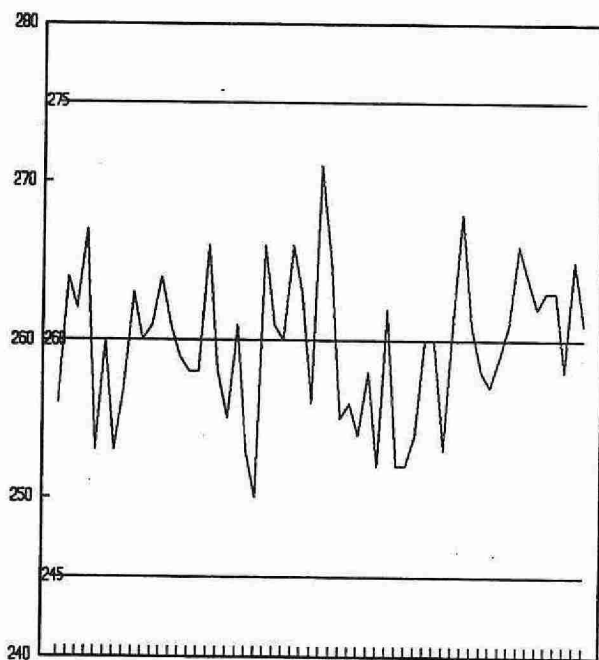
QUALITY CONTROL DATA FROM 13/01/94 TO 21/12/94



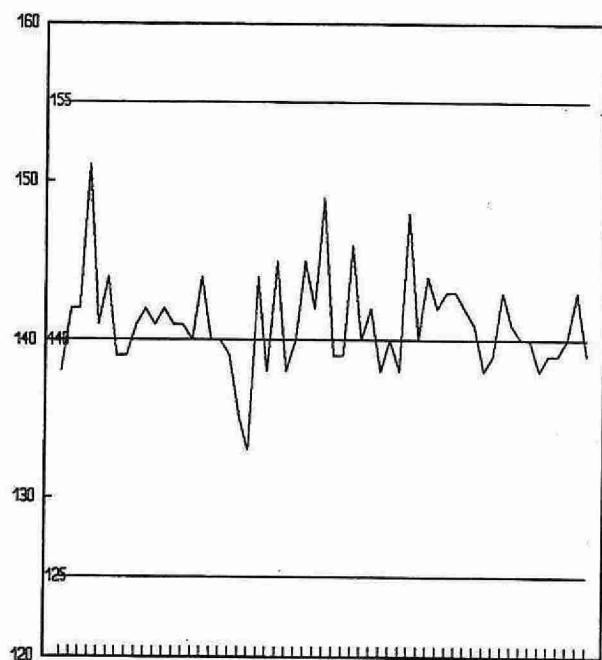
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## NITROGEN, NITRITE

### IDENTIFICATION:

|                     |  |                   |             |
|---------------------|--|-------------------|-------------|
| Laboratory Unit     | Colourimetry   | Method Introduced | 01/04/78    |
| Method Reference No | E3364A   | Units             | mg/L as N   |
| LIMS Product Code   | DISNUT3364   | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Rivers, Lakes, Precipitation, Soil Extracts, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride.

Approximate absorbance: 0.6 at the full scale level.

Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.001 | Current T value: 0.005 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|              |  |
|--------------|--|
| Calibration  | LTBL plus 3 standards, e.g. QCA  |
| Drift        | BL every 10 samples; standard every 20 samples   |
| Interference | Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression. |
| Recovery     | Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.              |

### NOTES:

Sept.'94 the method codes SDNP-E3223A, E3193A, E3184A and E3185 were amalgamated and a new method code SDNP-E3366A was generated.

# NITROGEN, NITRITE

QUALITY CONTROL DATA FROM 07/01/94 TO 04/12/94

Laboratory Unit: Colourimetry

Full Scale: to 0.200 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 94 | 0.160                  | 0.169              | 0.009     | 0.0009                 |
| B:   | 94 | 0.080                  | 0.079              | -0.001    | 0.0012                 |
| C:   | 94 | 0.016                  | 0.016              | 0.000     | 0.0007                 |
| A+B: | 94 | 0.240                  | 0.240              | 0.000     | 0.0014                 |
| A-B: | 94 | 0.080                  | 0.082              | 0.002     | 0.0015                 |
| B+C: | 94 | 0.096                  | 0.095              | -0.001    | 0.0016                 |
| B-C: | 94 | 0.064                  | 0.063              | -0.001    | 0.0012                 |

s.d.(AB) S(between runs): 0.0011

Sw(within run): 0.0011

S/Sw: 1.0

s.d.(BC) S(between runs): 0.0010

Sw(within run): 0.0008

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 0.231 | - | 0.249 | for | A+B |
| 0.074 | - | 0.086 | for | A-B |
| 0.092 | - | 0.100 | for | B+C |
| 0.061 | - | 0.067 | for | B-C |

## DUPLICATES:

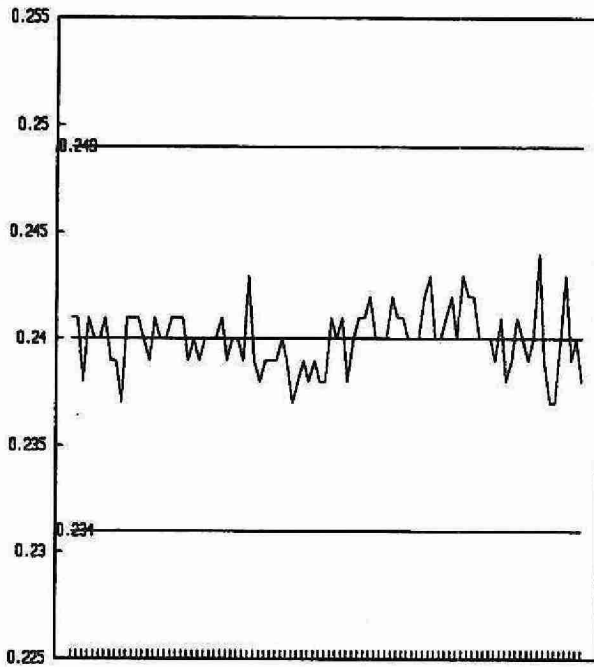
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 233          | 0.000 - 0.020             | 0.0014                 | 35.6                        |
| 28           | 0.021 - 0.040             | 0.0026                 | 11.6                        |
| 14           | 0.041 - 0.100             | 0.0053                 | 14.2                        |
| 3            | 0.101 - 0.200             | 0.0182                 | 11.2                        |
| 278          | Overall                   | 0.0017                 |                             |

## OTHER CHECKS:

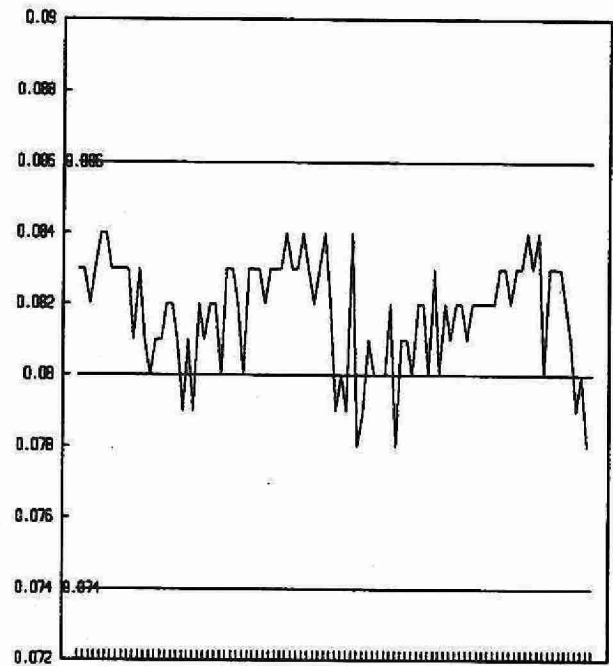
|                 | n  | Mean    | Standard Deviation (1) |
|-----------------|----|---------|------------------------|
| Long Term Blank | 94 | 0.00007 | 0.0006                 |

# NITROGEN, NITRITE (mg/L as N)

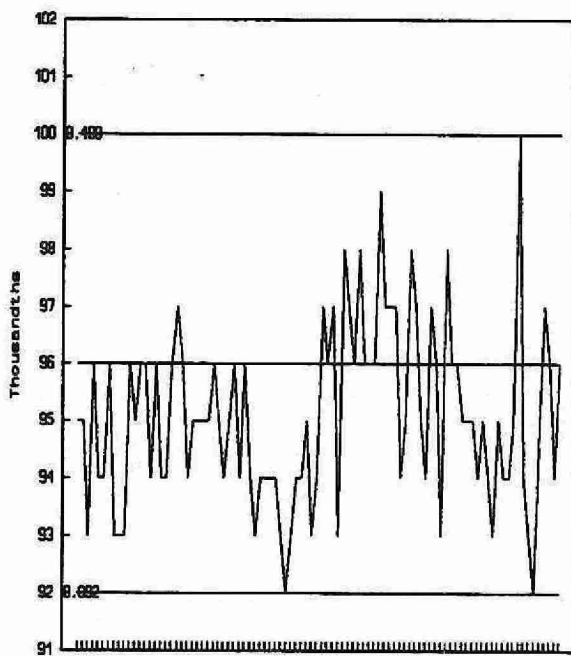
QUALITY CONTROL DATA FROM 07/01/94 TO 04/12/94



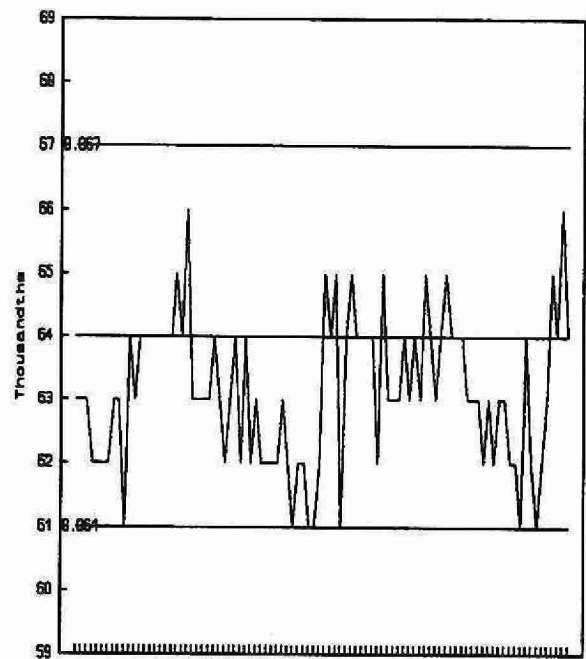
QUALITY CONTROL SAMPLE A+B



QUALITY CONTROL SAMPLE A-B



QUALITY CONTROL SAMPLE B+C



QUALITY CONTROL SAMPLE B-C

CONTROL LIMIT

## NITROGEN, NITRITE

### IDENTIFICATION:

|                     |   |                   |             |
|---------------------|---|-------------------|-------------|
| Laboratory Unit     | Colourimetry  | Method Introduced | 01/04/78    |
| Method Reference No | E3366A  | Units             | mg/L as N   |
| LIMS Product Code   | DISNUT3366  | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Sewage, Industrial Waste, Leachate, Domestic Waters |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride.

Approximate absorbance: 0.3 at the full scale level.

Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.005 | Current T value: 0.025 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|              |  |
|--------------|--|
| Calibration  | LTBL plus 3 standards, e.g. QCA  |
| Drift        | BL every 10 samples; standard every 20 samples   |
| Interference | Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression. |
| Recovery     | Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.              |

### NOTES:

Sept. '94 the method codes SDNP-E3223A, E3193A, E3184A and E3185 were amalgamated and a new method code SDNP-E3366A was generated.

# NITROGEN, NITRITE

QUALITY CONTROL DATA FROM 07/01/94 TO 20/12/94

Laboratory Unit: Colourimetry

Full Scale: to 2.00 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 83 | 1.60                      | 1.603                 | 0.003     | 0.0104                    |
| B:   | 83 | 0.80                      | 0.804                 | 0.004     | 0.0054                    |
| C:   | 83 | 0.160                     | 0.1601                | 0.0001    | 0.0016                    |
| A+B: | 83 | 2.40                      | 2.407                 | 0.007     | 0.0139                    |
| A-B: | 83 | 0.80                      | 0.799                 | -0.001    | 0.0091                    |
| B+C: | 83 | 0.960                     | 0.964                 | 0.004     | 0.0058                    |
| B-C: | 83 | 0.640                     | 0.644                 | 0.004     | 0.0055                    |

s.d.(AB) S(between runs): 0.008

Sw(within run): 0.006

S/Sw: 1.3

s.d.(BC) S(between runs): 0.004

Sw(within run): 0.004

S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 2.35  | - | 2.45  | for | A+B |
| 0.760 | - | 0.840 | for | A-B |
| 0.940 | - | 0.980 | for | B+C |
| 0.620 | - | 0.660 | for | B-C |

## DUPLICATES:

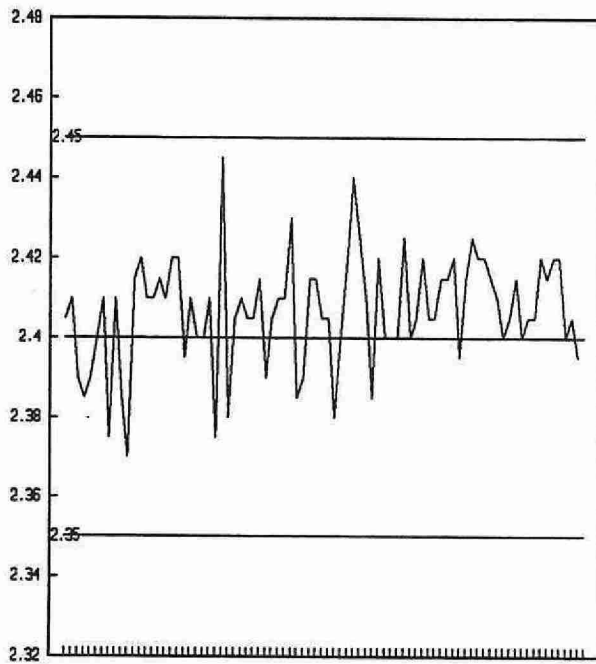
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 213             | 0.000 - 0.200                | 0.0030                    | 14.7                           |
| 12              | 0.201 - 0.400                | 0.0307                    | 9.7                            |
| 13              | 0.401 - 1.00                 | 0.0137                    | 2.3                            |
| 3               | 1.01 - 2.00                  | 0.0406                    | 2.5                            |
| 241             | Overall                      | 0.0089                    |                                |

## OTHER CHECKS:

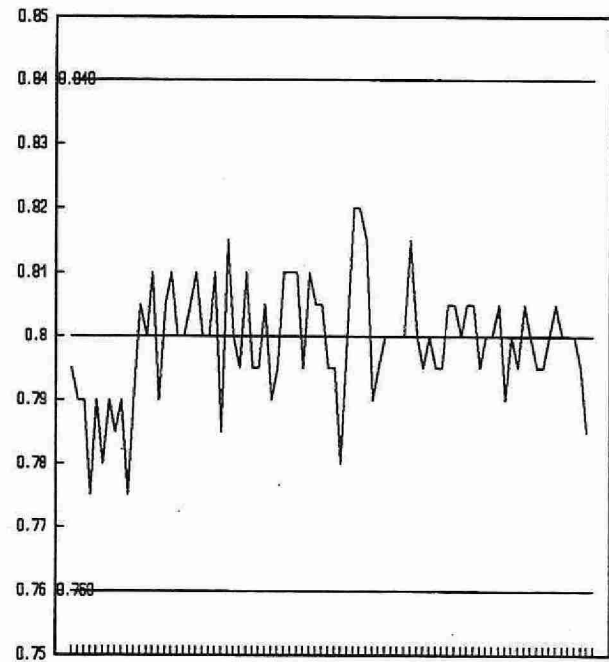
|                 | n  | Mean    | Standard<br>Deviation (1) |
|-----------------|----|---------|---------------------------|
| Long Term Blank | 83 | 0.00006 | 0.0012                    |

# NITROGEN, NITRITE (mg/L as N)

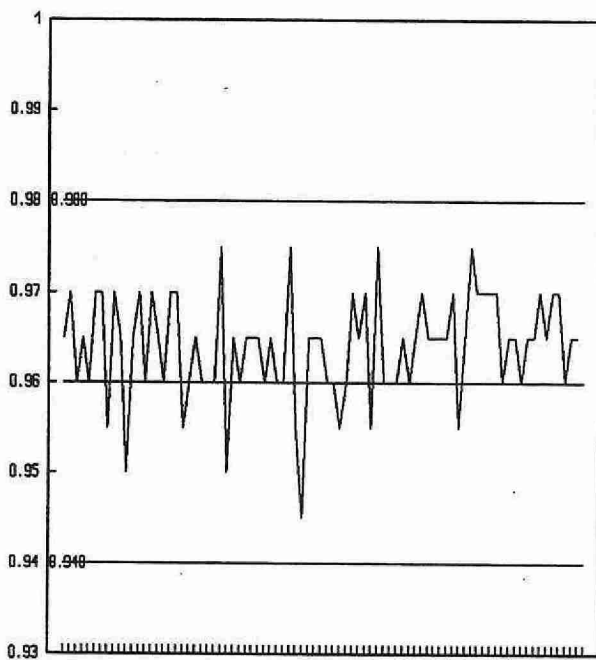
QUALITY CONTROL DATA FROM 07/01/94 TO 20/12/94



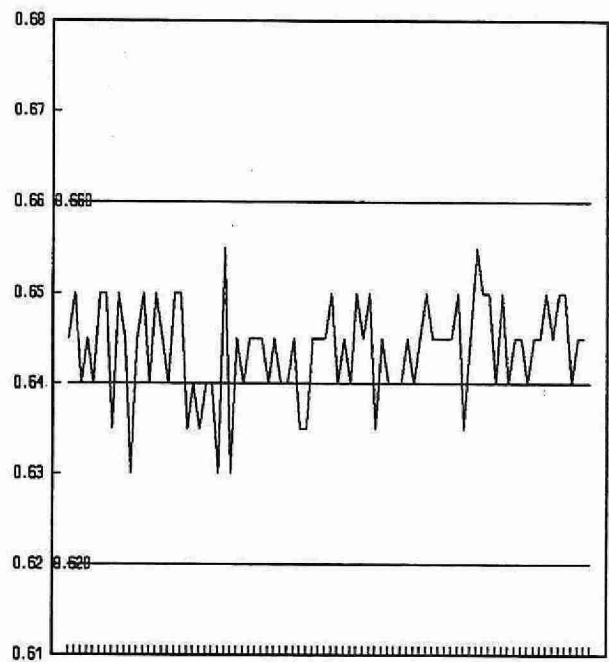
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT



## NITROGEN, TOTAL KJELDAHL

### IDENTIFICATION:

|                     |                   |                   |             |
|---------------------|-------------------|-------------------|-------------|
| Laboratory Unit     | Colourimetry      | Method Introduced | Mar '89     |
| Method Reference No | E3116A            | Units             | mg/g as N   |
| LIMS Product Code   | TNP3116           | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Soil and Sediment |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 0.08 to 0.4 g    |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Nitrogen compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

### INSTRUMENTATION:

Hot plate

Basic automated modular continuous flow system : 37.5°C bath. Colourimetric measurement is through a 5 cm. light path at 630 nm.

Data capture, and processing via a microcomputer system

### REPORTING:

#### SEDIMENT/SOIL

|   |                               |                               |
|---|-------------------------------|-------------------------------|
| Maximum Significant Figures: 2 decimal places | Current W value:<br>0.05/0.10 | Current T value:<br>0.25/0.50 |
|---|-------------------------------|-------------------------------|

### CALIBRATION:

3 High and 2 Low Calibration Standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | In house composite B-Soil/sediment, plus QC Soils/Sediment (RS92)       |
| Drift       | 4 BL's per run; high and low calibration standard at the end of the run |
| Recovery    | 1 digested BL plus 4 digested standards                                 |

### NOTES:

System is calibrated with undigested standards.

# NITROGEN, TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 07/01/94 TO 14/06/94

Laboratory Unit: Colourimetry

Full Scale: 0 to 20 mg/g as N

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| RS92 | 68 | 1.69                      | 1.693                 | 0.003     | 0.1003                    |

The calibration is accepted if the calibration control values obtained lie within the ranges:  
1.99 - 1.39 for RS92

## DUPLICATES: (Sediment)

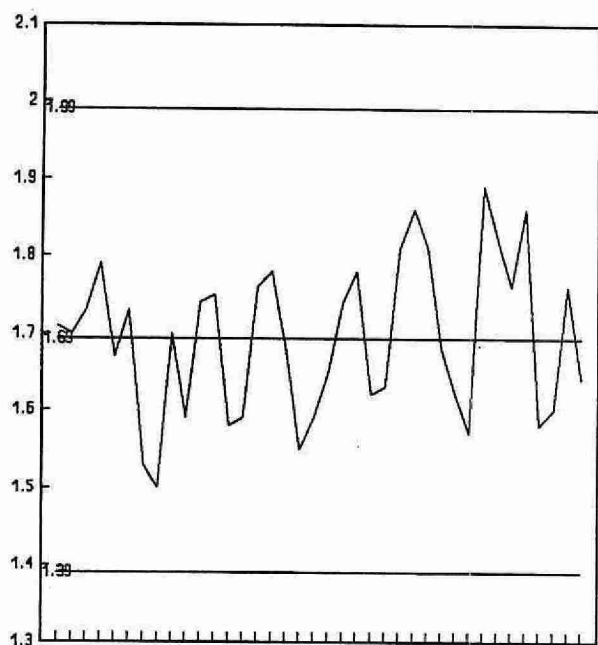
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 45              | 0.00 - 2.00                  | 0.0486                    | 4.5                            |
| 7               | 2.01 - 4.00                  | 0.1558                    | 5.4                            |
| 14              | 4.01 - 10.0                  | 0.2289                    | 3.4                            |
| 2               | 10.1 - 20.0                  | N.A.                      | N.A.                           |
| 68              | Overall                      | 0.0827                    |                                |

## DUPLICATES: (Soils)

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 15              | 0.00 - 2.00                  | 0.0756                    | 4.1                            |
| 46              | 2.01 - 4.00                  | 0.1864                    | 5.7                            |
| 14              | 4.01 - 10.0                  | 0.3368                    | 6.5                            |
| 1               | 10.1 - 20.0                  | N.A.                      | N.A.                           |
| 76              | Overall                      | 0.1951                    |                                |

NITROGEN, TOTAL KJELDAHL (mg/g as N)

QUALITY CONTROL DATA FROM 07/01/94 TO 14/07/94



QUALITY CONTROL STANDARD  
RS92 - Sediment and soil control

CONTROL LIMIT

## NITROGEN, TOTAL KJELDAHL

### IDENTIFICATION:

|                     |                                    |                   |             |
|---------------------|------------------------------------|-------------------|-------------|
| Laboratory Unit     | Colourimetry                       | Method Introduced | Mar '89     |
| Method Reference No | E3118A                             | Units             | mg/g as N   |
| LIMS Product Code   | TNP3118                            | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Terrestrial and aquatic vegetation |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 0.02 to 0.04 g   |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Nitrogen compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

### INSTRUMENTATION:

Hot plate

Basic automated modular continuous flow system : 37.5°C bath. Colourimetric measurement is through a 5 cm. light path at 630 nm.

Data capture, and processing via a microcomputer system

### REPORTING:

|   |                       |                       |
|---|-----------------------|-----------------------|
| Maximum Significant Figures: 2 decimal places | Current W value: 0.20 | Current T value: 1.00 |
|---|-----------------------|-----------------------|

### CALIBRATION:

3 High and 2 Low Calibration Standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | In house composite A-VEG, plus QC VEG (Pine Needles)                    |
| Drift       | 4 BL's per run; high and low calibration standard at the end of the run |
| Recovery    | 1 digested BL plus 4 digested standards                                 |

### NOTES:

System is calibrated with undigested standards.

# NITROGEN, TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 05/04/94 TO 17/10/94

Laboratory Unit: Colourimetry

Full Scale: to 100 mg/g as N

## CALIBRATION CONTROL:

|                             | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|-----------------------------|----|---------------------------|-----------------------|-----------|---------------------------|
| Pine Needles<br>(certified) | 20 | 12.10                     | 12.12                 | 0.02      | 0.6178                    |

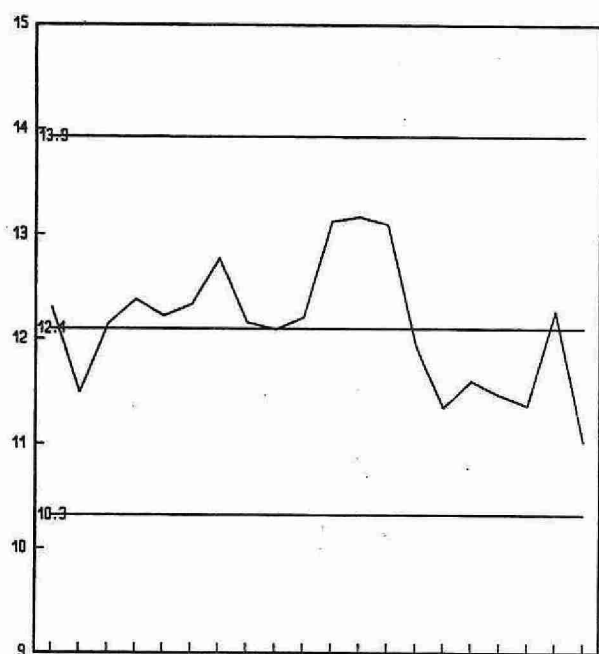
The calibration is accepted if the calibration control values obtained lie within the ranges:  
10.3 - 13.9 for Pine Needles

## DUPLICATES: (Vegetation)

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 0               | 0.0 - 5.0                    | N.A.                      | N.A.                           |
| 0               | 5.1 - 10.0                   | N.A.                      | N.A.                           |
| 37              | 10.1 - 25.0                  | 0.8737                    | 4.1                            |
| 17              | 25.1 - 50.0                  | 1.2173                    | 3.5                            |
| 54              | Overall                      | 0.9835                    |                                |

NITROGEN, TOTAL KJELDAHL (mg/g as N)

QUALITY CONTROL DATA FROM 05/04/94 TO 17/10/94



QUALITY CONTROL STANDARD  
(Pine needle)

CONTROL LIMIT

## NITROGEN, TOTAL KJELDAHL

### IDENTIFICATION:

|                     |  |                   |             |
|---------------------|--|-------------------|-------------|
| Laboratory Unit     | Colourimetry   | Method Introduced | 01/04/79    |
| Method Reference No | E3367A   | Units             | mg/L as N   |
| LIMS Product Code   | TOTNUT3367   | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Rivers, Lakes, Precipitation, Soil Extracts, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.3 at the full scale level.

Total phosphorus is determined simultaneously.

### INSTRUMENTATION:

Three block digesters

Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay).

Coulourimetric measurement is through a 5.0 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

### REPORTING:

|                                |                       |                      |
|--------------------------------|-----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.1 |
|--------------------------------|-----------------------|----------------------|

### CALIBRATION:

BL plus 7 undigested standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 undigested standards, e.g. QCA                    |
| Drift       | BL every 10 samples; undigested standard every 20 samples     |
| Recovery    | 3 digested BL plus 3 digested standards in duplicate, e.g. R1 |

# NITROGEN, TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 06/01/94 TO 28/12/94

Laboratory Unit: Colourimetry

Full Scale: to 2.00 mg/L as N

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 135 | 1.60                   | 1.61               | 0.01      | 0.0135                 |
| B:   | 135 | 0.800                  | 0.804              | 0.004     | 0.0089                 |
| C:   | 135 | 0.160                  | 0.163              | 0.003     | 0.0076                 |
| A+B: | 135 | 2.40                   | 2.42               | 0.02      | 0.0184                 |
| A-B: | 135 | 0.800                  | 0.807              | 0.007     | 0.0136                 |
| B+C: | 135 | 0.960                  | 0.967              | 0.007     | 0.0123                 |
| B-C: | 135 | 0.640                  | 0.642              | 0.002     | 0.0112                 |

|          |                  |       |                 |       |           |
|----------|------------------|-------|-----------------|-------|-----------|
| s.d.(AB) | S(between runs): | 0.011 | Sw(within run): | 0.010 | S/Sw: 1.2 |
| s.d.(BC) | S(between runs): | 0.008 | Sw(within run): | 0.008 | S/Sw: 1.0 |

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 2.32  | - | 2.48  | for | A+B |
| 0.740 | - | 0.860 | for | A-B |
| 0.910 | - | 1.01  | for | B+C |
| 0.610 | - | 0.670 | for | B-C |

## RECOVERIES:

| Number of Data | Expected Concentration | Mean Concentration | Standard Deviation (1) |
|----------------|------------------------|--------------------|------------------------|
| 135            | 1.40                   | 1.386              | 0.0418                 |
| 135            | 0.840                  | 0.830              | 0.0300                 |
| 135            | 0.280                  | 0.279              | 0.0173                 |

## DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 73           | 0.000 - 0.200             | 0.0172                 | 18.1                        |
| 165          | 0.201 - 0.400             | 0.0204                 | 9.4                         |
| 118          | 0.401 - 1.00              | 0.0336                 | 7.3                         |
| 40           | 1.01 - 2.00               | 0.0518                 | 15.7                        |
| 396          | Overall                   | 0.0266                 |                             |

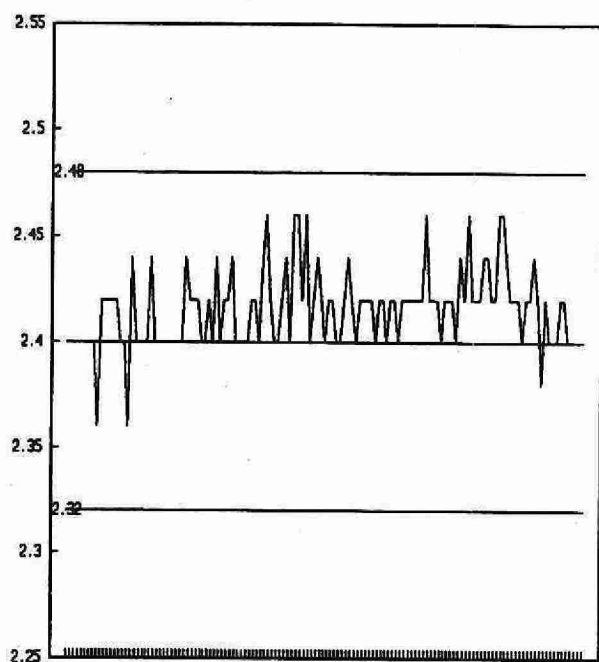
## OTHER CHECKS:

|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 135 | 0.0006 | 0.0077                 |
| Digested Blank  | 135 | 0.0130 | 0.0113                 |

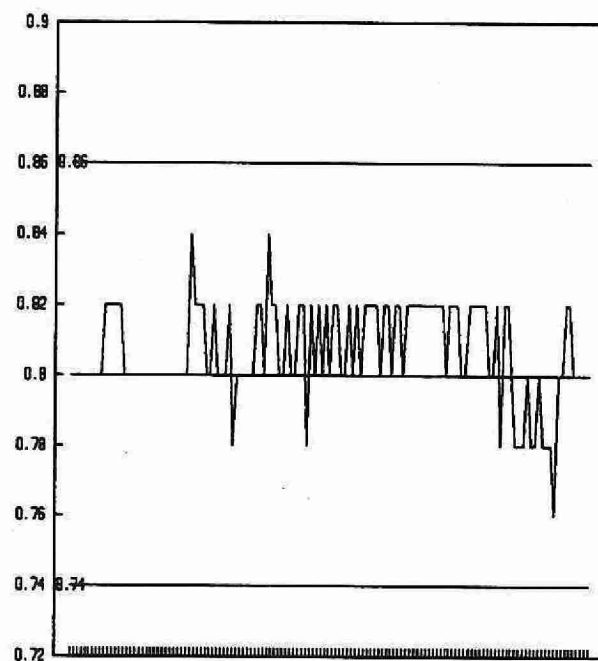


# NITROGEN, TOTAL KJELDAHL (mg/L as N)

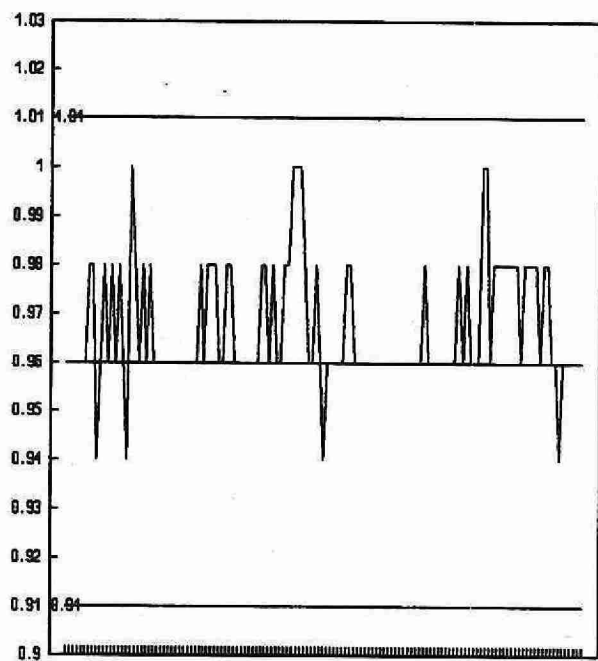
QUALITY CONTROL DATA FROM 06/01/94 TO 28/12/94



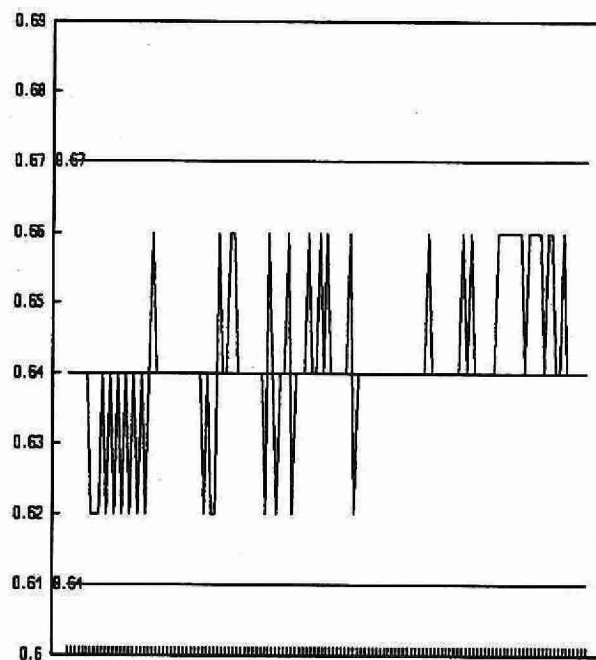
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## NITROGEN, TOTAL KJELDAHL

### IDENTIFICATION:

|                     |   |                   |             |
|---------------------|---|-------------------|-------------|
| Laboratory Unit     | Colourimetry  | Method Introduced | 01/04/79    |
| Method Reference No | E3368A  | Units             | mg/L as N   |
| LIMS Product Code   | TOTNUT3368  | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Sewage, Industrial Waste, Domestic Waters, Effluents, Leachates |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 1.1 at the full scale level.

Total phosphorus is determined simultaneously.

### INSTRUMENTATION:

Three block digesters

Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay).

Coulourimetric measurement is through a 1.5 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 undigested standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 undigested standards, e.g. QCA                    |
| Drift       | BL every 10 samples; undigested standard every 20 samples     |
| Recovery    | 3 digested BL plus 3 digested standards in duplicate, e.g. R1 |

### NOTES:

System is calibrated with undigested standards.

# NITROGEN, TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 05/01/94 TO 20/12/94

Laboratory Unit: Colourimetry

Full Scale: to 50.0 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 84 | 40.0                   | 40.048             | -0.048    | 0.1632                 |
| B:   | 84 | 20.0                   | 20.062             | -0.062    | 0.1040                 |
| C:   | 84 | 4.0                    | 4.019              | -0.019    | 0.0465                 |
| A+B: | 84 | 60.0                   | 60.110             | -0.110    | 0.2242                 |
| A-B: | 84 | 20.0                   | 19.986             | 0.014     | 0.1569                 |
| B+C: | 84 | 24.0                   | 24.081             | -0.081    | 0.1322                 |
| B-C: | 84 | 16.0                   | 16.043             | -0.043    | 0.0922                 |

s.d.(AB) S(between runs): 0.1368 Sw(within run): 0.1109 S/Sw: 1.2  
s.d.(BC) S(between runs): 0.0806 Sw(within run): 0.0652 S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

59.3 - 60.7 for A+B  
19.4 - 20.6 for A-B  
23.6 - 24.4 for B+C  
15.7 - 16.3 for B-C

## RECOVERIES:

| Number of Data | Expected Concentration | Mean Concentration | Standard Deviation (1) |
|----------------|------------------------|--------------------|------------------------|
| 84             | 35                     | 34.87              | 0.5747                 |
| 84             | 21                     | 20.88              | 0.3187                 |
| 84             | 7                      | 6.92               | 0.1960                 |

## DUPLICATES:

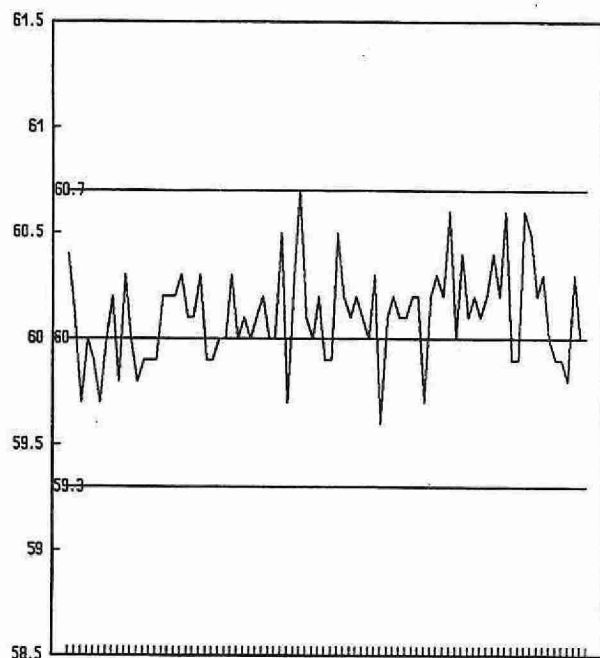
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 130          | 0.00 - 5.00               | 0.0643                 | 12.2                        |
| 17           | 5.01 - 10.0               | 0.3851                 | 7.6                         |
| 55           | 10.1 - 25.0               | 0.4180                 | 15.3                        |
| 38           | 25.1 - 50.0               | 0.7234                 | 2.6                         |
| 240          | Overall                   | 0.2537                 |                             |

## OTHER CHECKS:

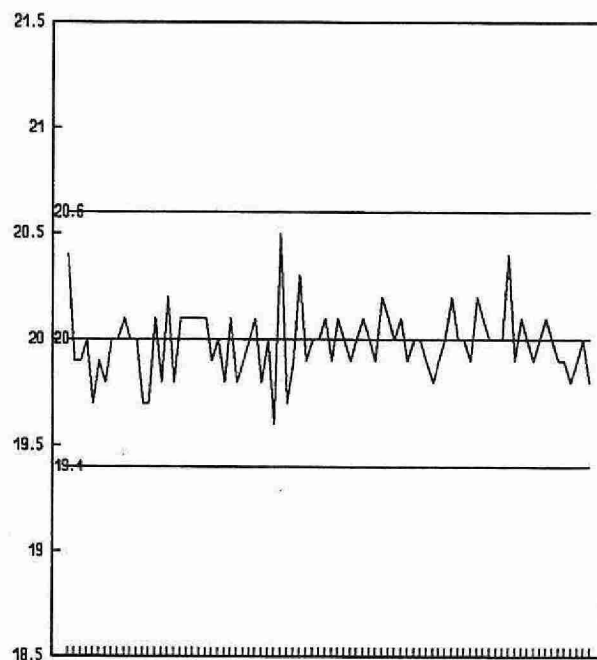
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 84 | 0.020 | 0.0381                 |
| Digested Blank  | 84 | 0.032 | 0.0584                 |

# NITROGEN, TOTAL KJELDAHL (mg/L as N)

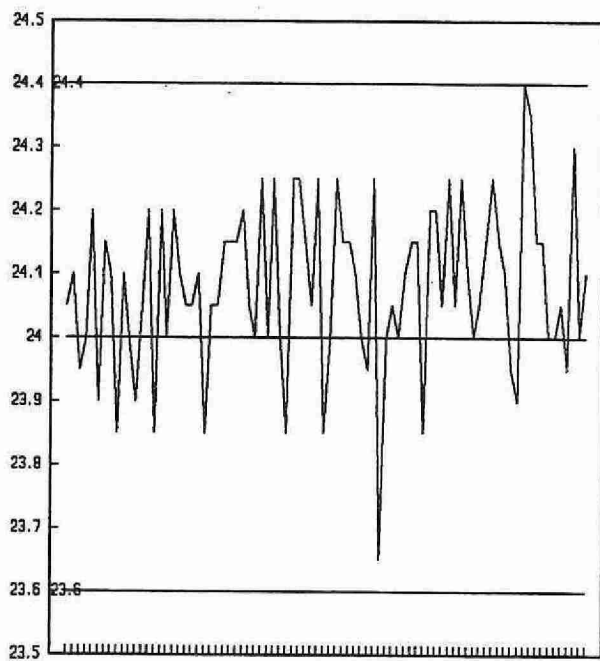
QUALITY CONTROL DATA FROM 05/01/94 TO 12/12/94



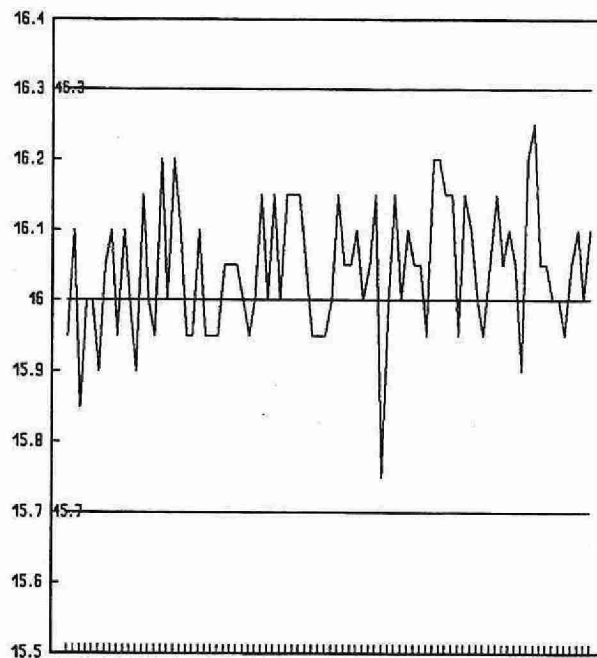
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## OXYGEN DEMAND, BIOCHEMICAL

### IDENTIFICATION:

|                      |   |                    |            |
|----------------------|---|--------------------|------------|
| Laboratory Unit:     | BOD   | Method Introduced: | Before '61 |
| Method Reference No: | E3182A  | Units:             | mg/L as O  |
| LIMS Product Code:   | BOD3182   | Supervisor:        | F. Lo      |
| Sample Type/Matrix:  | Sewage, Industrial Waste, Effluents, Domestic Waters, Leachates |                    |            |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: | 400 mL           |
| Container:         | Glass or plastic |

### SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

### ANALYTICAL PROCEDURE:

Oxygen depletion is measured as the difference in dissolved oxygen (DO) concentration. DO readings are taken prior to sample storage, and also at the end of storage in the dark at 20°C for five days (BOD<sub>5</sub>). If necessary, dilutions are made with aerated, nutrient-enriched water to obtain a 25-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples, calculation of an appropriate seed correction is required.

### INSTRUMENTATION:

- YSI Model 59 DO meter (Yellow Springs Instrument Company) with DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen (1 mil = 0.001 inch).
- Titration equipment for Winkler analysis of dissolved oxygen.
- Incubator (19-21°C); BOD bottles (300 mL)

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION (DO):

The standard is air-saturated reversed osmosis deionized water. The DO content is read from a table (ORBISPHERE LABORATORIES - Pressure temperature dissolved oxygen table) after measuring its temperature and the barometric pressure in the laboratory.

## OXYGEN DEMAND, BIOCHEMICAL cont'd

### CONTROLS:

|                  |   |
|------------------|---|
| Calibration (DO) | 2 QC solutions of Pure-DW water which have been partially stripped of DO by flushing with nitrogen. These "solutions", of different but unknown DO, are compared using the Oxygen meter and the Winkler titration procedure. The difference between the values for the two analytical methods is utilized as a slope control for the DO Analyzer. |
| Recovery (BOD5)* | 3 Recovery standards prepared from a combination of Glucose and Glutamic Acid e.g. R1; the expected BOD5 is 67% of the oxygen requirement for complete oxidation.   |
| Drift            | Air saturated Pure-DW water after every 24 samples.   |
| Blanks*          | Pure-DW water and BOD dilution water  |

### NOTES:

\* These solutions are incubated for five days alongside samples.

# OXYGEN DEMAND, BIOCHEMICAL

QUALITY CONTROL DATA FROM 07/01/94 TO 30/12/94

Laboratory Unit: BOD

Full Scale: to 9.0 mg/L as O at 20°C

## CALIBRATION CONTROL:

|    | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|----|-----|------------------------|--------------------|-----------|------------------------|
| A: | 101 | 0.00                   | 0.026              | 0.026     | 0.0929                 |
| B: | 101 | 0.00                   | 0.013              | 0.013     | 0.0821                 |

On any given day the calibration is accepted if the values obtained lie within the ranges:

-0.25 - 0.25

## RECOVERIES:

| Number of Data | Expected Depletion | Mean Depletion | Standard Deviation (1) |
|----------------|--------------------|----------------|------------------------|
| 51             | 2.20               | 2.15           | 0.1937                 |
| 51             | 4.34               | 4.25           | 0.2367                 |
| 51             | 6.52               | 6.38           | 0.2545                 |

## DUPLICATES:

| n Data Pairs | Sample Depletion Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|-----------------------|------------------------|-----------------------------|
| 49           | 0.0 - 1.8             | 0.0299                 | 3.2                         |
| 52           | 1.9 - 4.5             | 0.0414                 | 3.9                         |
| 35           | 4.6 - 9.0             | 0.0617                 | 1.8                         |
| 136          | Overall               | 0.0400                 |                             |

## OTHER CHECKS:

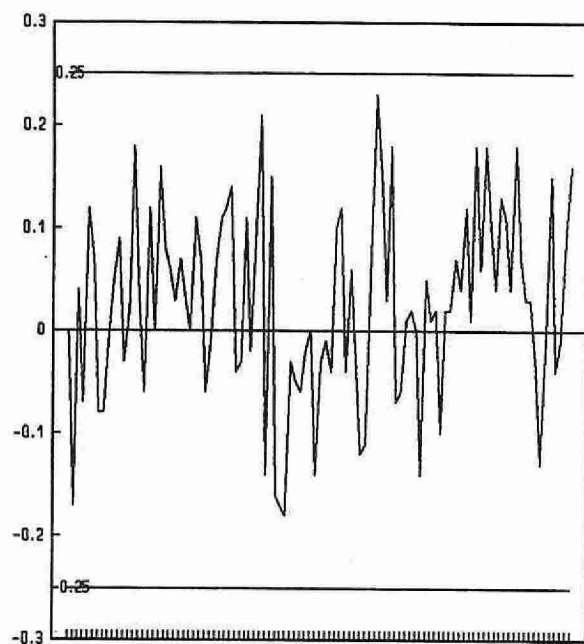
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| 5 Day DPW Blank | 52 | 0.128 | 0.0978                 |
| 5 Day BOD Blank | 52 | 0.149 | 0.1117                 |

## NOTES:

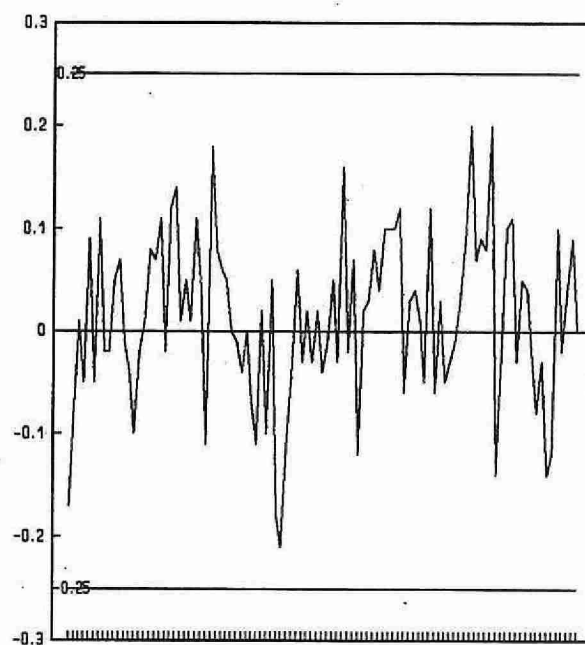
The final concentration of BOD in mg/L as O is determined by the oxygen depletion after 5 days at 20°C multiplied by a dilution and seed correction factor.

**OXYGEN DEMAND, BIOCHEMICAL** (mg/L as O)

QUALITY CONTROL DATA FROM 07/01/94 TO 30/12/94



QUALITY CONTROL STANDARD A



QUALITY CONTROL STANDARD B

CONTROL LIMIT



## OXYGEN DEMAND, CHEMICAL

### IDENTIFICATION:

|                     |                                       |                   |             |
|---------------------|---------------------------------------|-------------------|-------------|
| Laboratory Unit     | Colourimetry                          | Method Introduced | 01/07/82    |
| Method Reference No | E3170A                                | Units             | mg/L as O   |
| LIMS Product Code   | COD3170                               | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Domestic Waters, Leachates, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 25 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 149°C. Analysis is completed by automated colourimetric measurement of trivalent chromium. Approximate absorbance: 0.05 at the full scale level.

### INSTRUMENTATION:

- Culture tubes with Teflon closures; mechanical-convection oven
- Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

### REPORTING:

|                                |                    |                    |
|--------------------------------|--------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 1 | Current T value: 5 |
|--------------------------------|--------------------|--------------------|

### CALIBRATION:

3 digested BL plus 3 digested standards

### CONTROLS:

|              |  |
|--------------|--|
| Calibration  | 2 digested standards, e.g. QCA   |
| Drift        | Undigested BL every 10 samples; standard plus BL at end of run   |
| Recovery     | 2 digested standards, e.g. R1  |
| Interference | Digested standard (40 mg/L as O) spiked with 50 mg/L Cl confirms suppression of chloride interference. |

### NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week. The recovery standard is a material known to be very difficult to digest. The expected recovery is approximately 85%, based on long term experience. We continue to use this material in spite of the poor recovery, because if the slightest problem exists with the digestion step, the recovery falls off sharply to approximately 10%.

# OXYGEN DEMAND, CHEMICAL

QUALITY CONTROL DATA FROM 07/01/94 TO 23/12/94

Laboratory Unit: Colourimetry

Full Scale: to 40.0 mg/L as O

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 53 | 40.0                   | 39.89              | 0.113     | 0.6881                 |
| B:   | 53 | 10.0                   | 9.87               | 0.132     | 0.8327                 |
| A+B: | 53 | 50.0                   | 49.76              | 0.245     | 1.1479                 |
| A-B: | 53 | 30.0                   | 30.02              | -0.018    | 1.0080                 |

s.d.(AB)

S(between runs): 0.76

Sw(within run): 0.71

S/Sw: 1.1

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

46.3 - 53.7 for A+B

27.2 - 32.8 for A-B

## RECOVERIES:

| Number of Data | Expected Concentration | Mean Concentration | Standard Deviation (1) |
|----------------|------------------------|--------------------|------------------------|
| 53             | 39                     | 38.46              | 1.3248                 |
| 53             | 9.8                    | 9.12               | 0.9800                 |

## DUPLICATES:

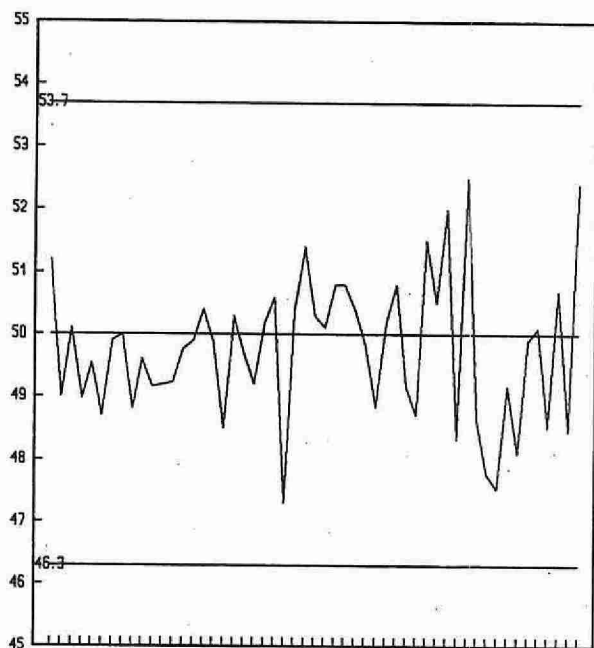
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 17           | 0 - 8                     | 0.0805                 | 16.6                        |
| 74           | 9 - 20                    | 1.5429                 | 10.3                        |
| 48           | 21 - 40                   | 1.9331                 | 5.8                         |
| 139          | Overall                   | 1.6132                 |                             |

## OTHER CHECKS:

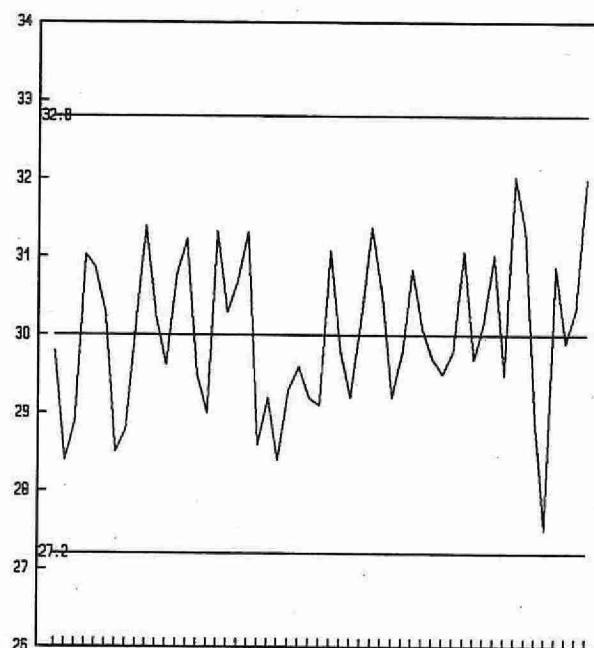
|                | n  | Mean   | Standard Deviation (1) |
|----------------|----|--------|------------------------|
| Chloride Check | 40 | 38.832 | 1.4706                 |

OXYGEN DEMAND, CHEMICAL (mg/L as O)

QUALITY CONTROL DATA FROM 07/01/94 TO 23/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## OXYGEN DEMAND, CHEMICAL

### IDENTIFICATION:

|                     |   |                   |             |
|---------------------|---|-------------------|-------------|
| Laboratory Unit     | Colourimetry  | Method Introduced | 01/07/82    |
| Method Reference No | E3246A  | Units             | mg/L as O   |
| LIMS Product Code   | COD3246   | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Sewage, Industrial Waste, Domestic Waters, Leachates, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 25 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 149°C. Analysis is completed by automated colourimetric measurement of trivalent chromium. Approximate absorbance: 0.6 at the full scale level.

### INSTRUMENTATION:

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |
|--------------------------------|--------------------|---------------------|

### CALIBRATION:

2 digested BL plus 4 digested standards

### CONTROLS:

|              |   |
|--------------|---|
| Calibration  | 2 digested standards, e.g. QCA  |
| Drift        | Undigested BL every 10 samples; standard plus BL at end of run  |
| Recovery     | 2 digested standards, e.g. R1   |
| Interference | Digested standard (50 mg/L as O) spiked with 900 mg/L Cl confirms suppression of chloride interference. |

### NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week. The recovery standard is a material known to be very difficult to digest. The expected recovery is approximately 85%, based on long term experience. We continue to use this material in spite of the poor recovery, because if the slightest problem exists with the digestion step, the recovery falls off sharply to approximately 10%.

# OXYGEN DEMAND, CHEMICAL

QUALITY CONTROL DATA FROM 17/01/94 TO 23/12/94

Laboratory Unit: Colourimetry

Full Scale: to 500.0 mg/L as O

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 53 | 400                    | 397.6              | 2.434     | 4.4515                 |
| B:   | 53 | 100                    | 101.8              | -1.849    | 3.3091                 |
| A+B: | 53 | 500                    | 499.4              | 0.585     | 6.2951                 |
| A-B: | 53 | 300                    | 295.7              | 4.283     | 4.6802                 |

s.d.(AB)

S(between runs): 3.92

Sw(within run): 3.31

S/Sw: 1.2

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

477.5 - 522.5 for A+B  
285.0 - 315.0 for A-B

## RECOVERIES:

| Number of Data | Expected Concentration | Mean Concentration | Standard Deviation (1) |
|----------------|------------------------|--------------------|------------------------|
| 53             | 390                    | 390.7              | 4.6469                 |
| 53             | 98                     | 100.4              | 4.7180                 |

## DUPLICATES:

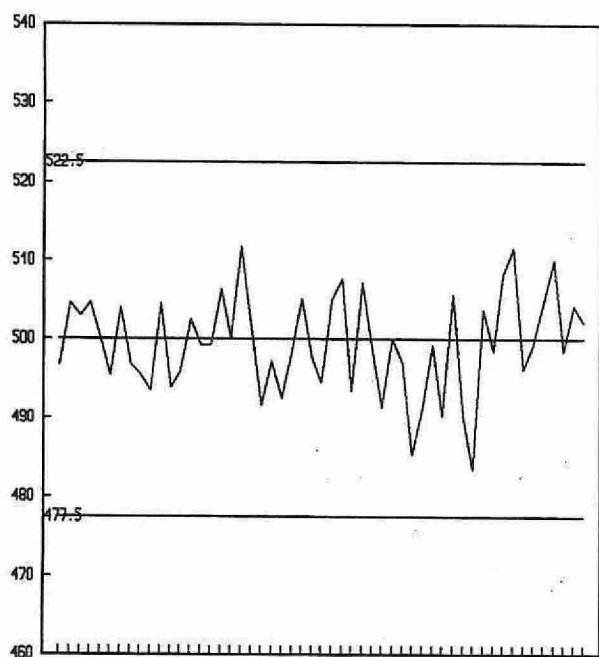
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 59           | 0 - 50                    | 3.0403                 | 9.9                         |
| 26           | 51 - 100                  | 3.3983                 | 4.3                         |
| 13           | 101 - 250                 | 5.6312                 | 3.5                         |
| 15           | 251 - 500                 | 11.6321                | 2.9                         |
| 113          | Overall                   | 4.0978                 |                             |

## OTHER CHECKS:

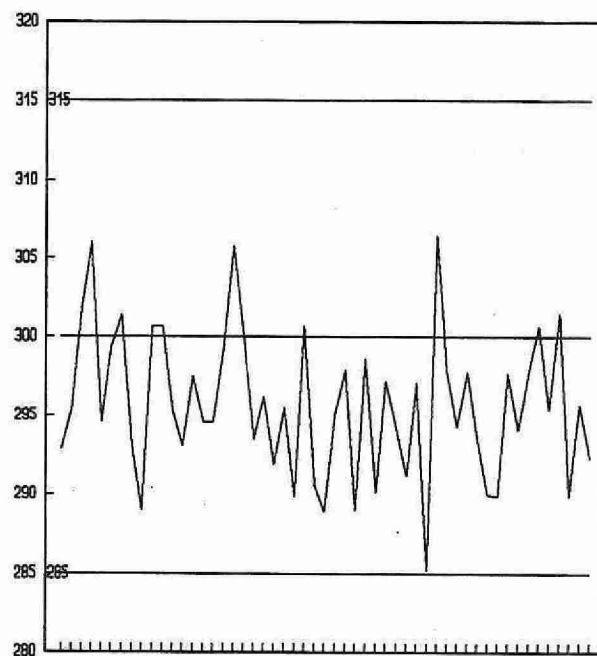
|                | n  | Mean | Standard Deviation (1) |
|----------------|----|------|------------------------|
| Chloride Check | 53 | 54.4 | 4.5240                 |

# OXYGEN DEMAND, CHEMICAL (mg/L as O)

QUALITY CONTROL DATA FROM 17/01/94 TO 23/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## pH

### IDENTIFICATION:

|                      |   |                   |               |
|----------------------|---|-------------------|---------------|
| Laboratory Unit      | Dorset  | Method Introduced | 01/01/76      |
| LIS Test Name Code   | pH  | Units             | dimensionless |
| Work Station Code    | DOT   | Unit Code         | nil           |
| Method Code          | 0902PH  | Supervisor        | J. McBride    |
| Method Reference No. | E3042A  |                   |               |
| Sample Type/Matrix:  | Streams, Lakes, Precipitation, and Groundwaters |                   |               |

### SAMPLING:

|                    |   |
|--------------------|---|
| Quantity Required: | 150 mL  |
| Container:         | 250 mL Amber polyethylene or BOD bottle filled to the brim; screw caps with cone-shaped liners are preferred. |

### ANALYTICAL PROCEDURE:

pH is measured directly on a stirred sample (100 mL) at room temperature. Stirring rate, beaker size, degree of electrode immersion and room temperature range are uniform for all samples and standards. Alkalinity (Gran) is performed simultaneously.

### INSTRUMENTATION:

Digital pH meter, stirrer, combined glass electrode.

### REPORTING:

Maximum Significant Figures: 3

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

### CONTROLS:

|             |                                    |
|-------------|------------------------------------|
| Calibration | BL plus 2 standards, e.g. QCA      |
| Drift       | 2 standard buffers - 2 times daily |

# pH

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94

Laboratory : Dorset

Analytical Range: to 14.00 Dimensionless

## CALIBRATION CONTROL:

|      | n   | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|-----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 128 | 6.86                      | 6.87                  | 0.01      | 0.0139                    |
| B:   | 128 | 4.01                      | 4.01                  | 0.003     | 0.0154                    |
| A+B: | 128 | 10.87                     | 10.88                 | 0.01      | 0.0178                    |
| A-B: | 128 | 2.85                      | 2.86                  | 0.01      | 0.0232                    |

s.d.(AB)

S(between runs): 0.015

S/Sw:(within run): 0.016

S/Sw: 0.89

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.65 - 11.07 for A+B  
2.72 - 3.00 for A-B

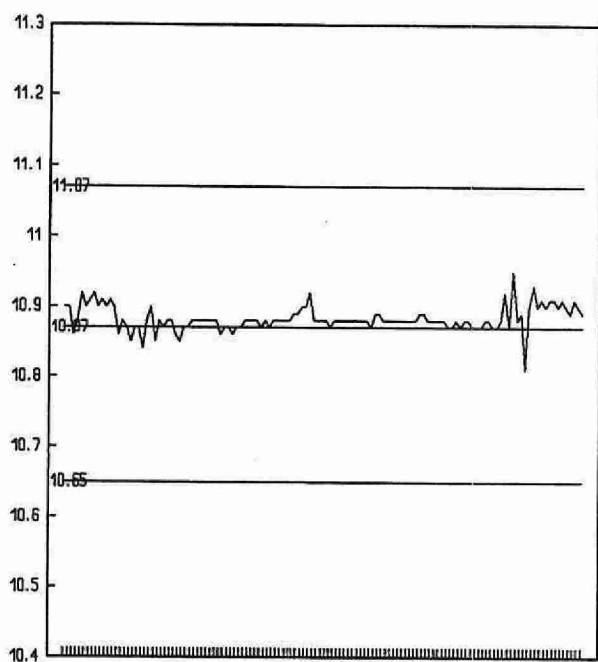
## DUPLICATES:

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 80              | 3.5 - 5.00                   | 0.0212                    | 0.5                            |
| 118             | 5.01 - 6.00                  | 0.0306                    | 0.6                            |
| 122             | 6.01 - 7.00                  | 0.0320                    | 0.6                            |
| 55              | 7.01 - 9.00                  | 0.0206                    | 0.3                            |
| 375             | Overall                      | 0.0279                    |                                |

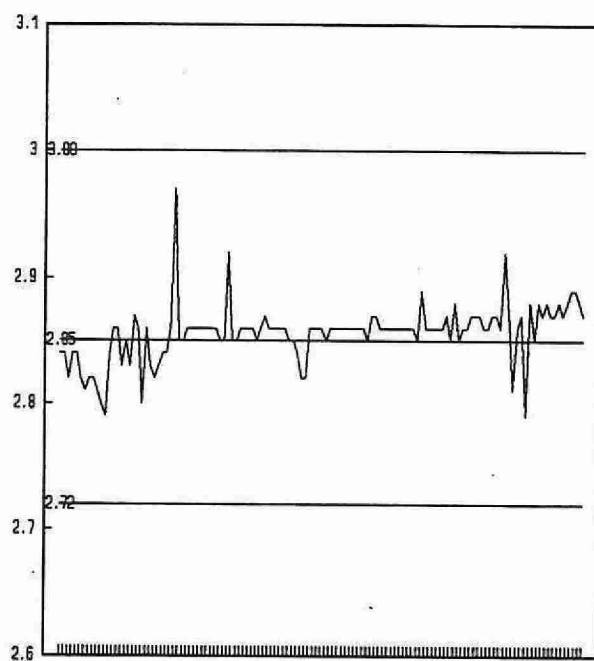


pH

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

pH

**IDENTIFICATION:**

|                     |                                    |                   |               |
|---------------------|------------------------------------|-------------------|---------------|
| Laboratory Unit     | Titration                          | Method Introduced | 09/07/80      |
| Method Reference No | E3218A                             | Units             | Dimensionless |
| LIMS Product Code   | PHALCO3218, CONDPH3218             | Supervisor        | F. Lo         |
| Sample Type/Matrix  | Domestic Waters, Sewage, Effluents |                   |               |

**SAMPLING:**

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or Plastic |

**ANALYTICAL PROCEDURE:**

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Total fixed endpoint alkalinity, and conductivity are determined simultaneously.

**INSTRUMENTATION:**

Automated modular titration system with microcomputer control and data processing software.

**REPORTING:**

Maximum Significant Figures: 3

**CALIBRATION:**

2 standard buffers covering the pH range of 4 to 9

**CONTROLS:**

|             |   |
|-------------|---|
| Calibration | 2 QC standards e.g. QCA   |
| Drift       | In run standards throughout the run (diluted tap water 50% V/V) |

# pH

QUALITY CONTROL DATA FROM 04/01/94 TO 21/12/94

Laboratory Unit: Titration

Analytical Range: to 14.00 Dimensionless

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 98 | 7.41                      | 7.418                 | 0.008     | 0.0122                    |
| B:   | 98 | 4.45                      | 4.475                 | 0.025     | 0.0295                    |
| A+B: | 98 | 11.86                     | 11.893                | 0.033     | 0.0336                    |
| A-B: | 98 | 2.96                      | 2.943                 | -0.017    | 0.0301                    |

s.d.(AB)      S(between runs): 0.023      Sw(within run): 0.021      S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

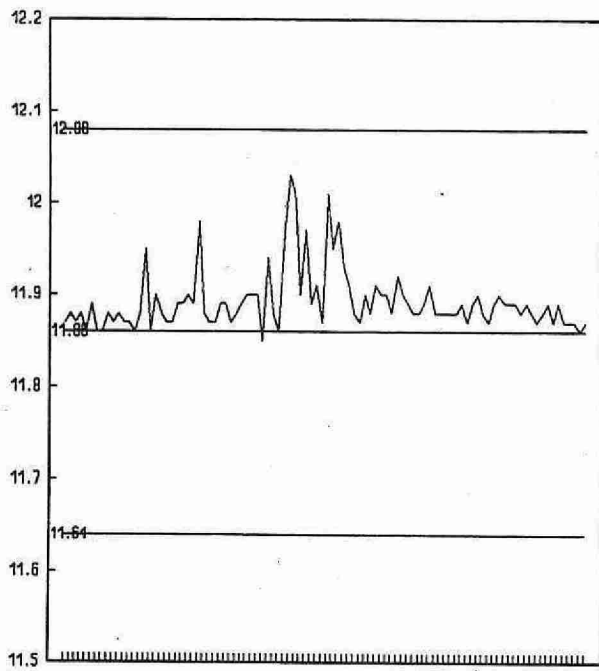
11.64 - 12.08 for A+B  
2.79 - 3.13 for A-B

## DUPLICATES:

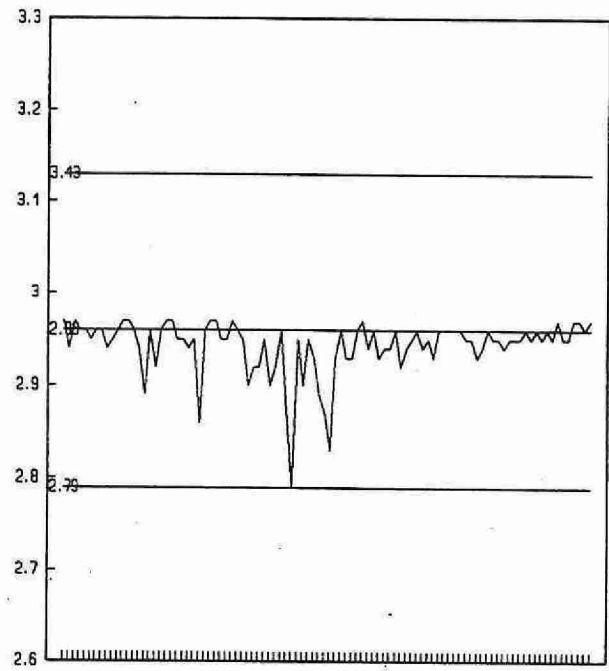
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 15              | 1.00 - 7.00                  | 0.1209                    | 1.9                            |
| 132             | 7.01 - 8.00                  | 0.1603                    | 1.9                            |
| 77              | 8.01 - 12.00                 | 0.0816                    | 1.0                            |
| 224             | Overall                      | 0.1329                    |                                |

pH

QUALITY CONTROL DATA FROM 04/01/94 TO 21/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## pH

### IDENTIFICATION:

|                     |                    |                   |               |
|---------------------|--------------------|-------------------|---------------|
| Laboratory Unit     | Titration          | Method Introduced | before '70    |
| Method Reference No | E3228A             | Units             | Dimensionless |
| LIMS Product Code   | PHALK3228, PH3228  | Supervisor        | F. Lo         |
| Sample Type/Matrix  | Landfill leachates |                   |               |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 15 mL            |
| Container         | Glass or Plastic |

### ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (15.0 mL) at room temperature. Stirring rate and room temperature range are uniform for all samples and standards.

### INSTRUMENTATION:

pH meter, stirrer, Radiometer combination electrode.

### REPORTING:

Maximum Significant Figures: 3

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

### CONTROLS:

|             |                         |
|-------------|-------------------------|
| Calibration | 2 QC standards e.g. QCA |
|-------------|-------------------------|

# pH

QUALITY CONTROL DATA FROM 22/02/94 TO 29/12/94

Laboratory Unit: Titration

Analytical Range: to 14.00 Dimensionless

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 22 | 7.41                      | 7.401                 | -0.009    | 0.0173                    |
| B:   | 22 | 4.45                      | 4.442                 | -0.008    | 0.0242                    |
| A+B: | 22 | 11.86                     | 11.843                | -0.017    | 0.0329                    |
| A-B: | 22 | 2.96                      | 2.9595                | 0.0005    | 0.0263                    |

s.d.(AB)    S(between runs): 0.021    Sw(within run): 0.019    S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

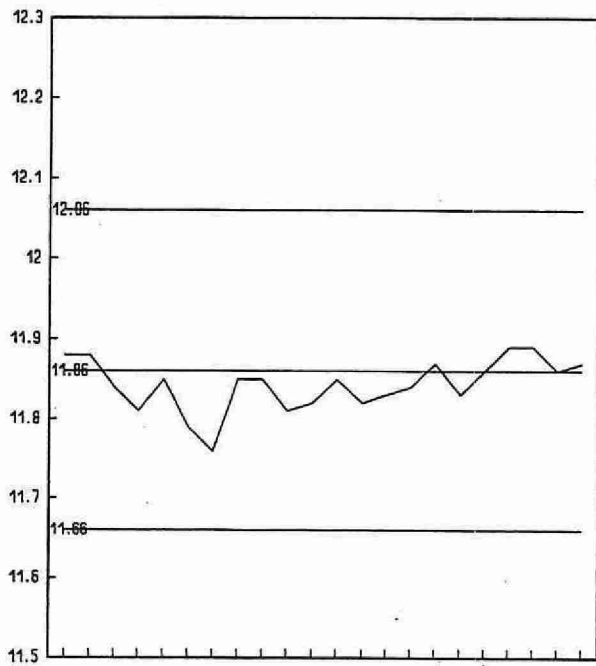
11.66 - 12.06 for A+B  
2.81 - 3.11 for A-B

## DUPLICATES:

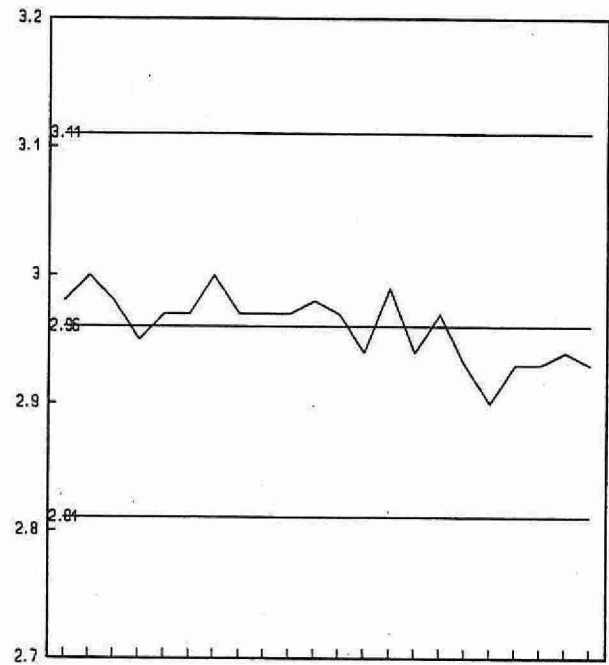
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 48              | 4.00 - 8.00                  | 0.0271                    | 0.5                            |
| 8               | 8.01 - 8.50                  | 0.0264                    | 0.3                            |
| 1               | 8.51 - 14.00                 | N.A.                      | N.A.                           |
| 57              | Overall                      | 0.0263                    |                                |

pH

QUALITY CONTROL DATA FROM 22/02/94 TO 29/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

**pH**

**IDENTIFICATION:**

|                     |                                      |                   |               |
|---------------------|--------------------------------------|-------------------|---------------|
| Laboratory Unit     | Titration                            | Method Introduced | 01/05/79      |
| Method Reference No | E3248A                               | Units             | Dimensionless |
| LIMS Product Code   | PHACD3248,PH3248                     | Supervisor        | F. Lo         |
| Sample Type/Matrix  | Precipitation, Throughfall, Stemflow |                   |               |

**SAMPLING:**

|                   |                  |
|-------------------|------------------|
| Quantity Required | 15 mL            |
| Container         | Glass or Plastic |

**ANALYTICAL PROCEDURE:**

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Total fixed endpoint acidity and Gran acidity are determined simultaneously.

**INSTRUMENTATION:**

Automated modular titration system with microcomputer control and data processing software.

**REPORTING:**

Maximum Significant Figures: 3

**CALIBRATION:**

2 standard buffers covering the pH range of 4 to 9

**CONTROLS:**

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
|-------------|---------------------------------|



# pH

QUALITY CONTROL DATA FROM 10/01/94 TO 14/12/94

Laboratory Unit: Titration

Analytical Range: to 14.00 Dimensionless

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 29 | 4.45                      | 4.447                 | -0.003    | 0.0089                    |
| B:   | 29 | 3.75                      | 3.740                 | -0.010    | 0.0130                    |
| A+B: | 29 | 8.20                      | 8.187                 | -0.013    | 0.0203                    |
| A-B: | 29 | 0.70                      | 0.707                 | 0.007     | 0.0090                    |

s.d.(AB) S(between runs): 0.011

S/Sw:(within run): 0.006

S/Sw: 1.8

On any given day the calibration is accepted if the values obtained lie within the ranges:

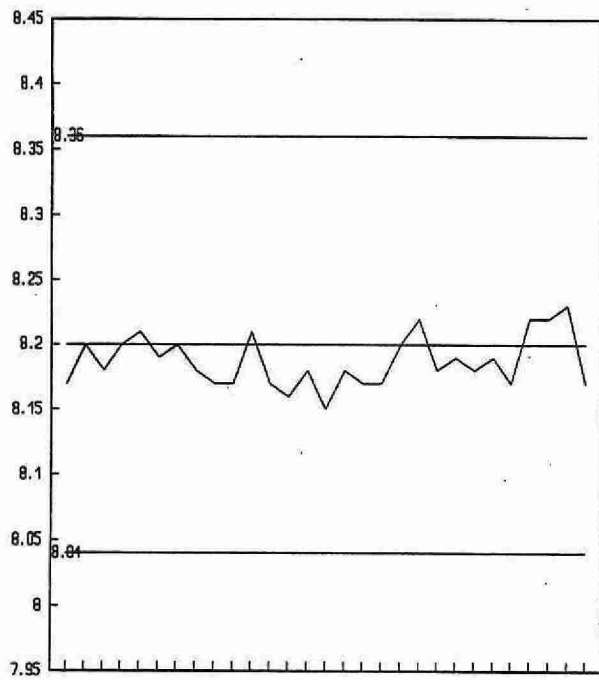
8.04 - 8.36 for A+B  
0.59 - 0.81 for A-B

## DUPLICATES:

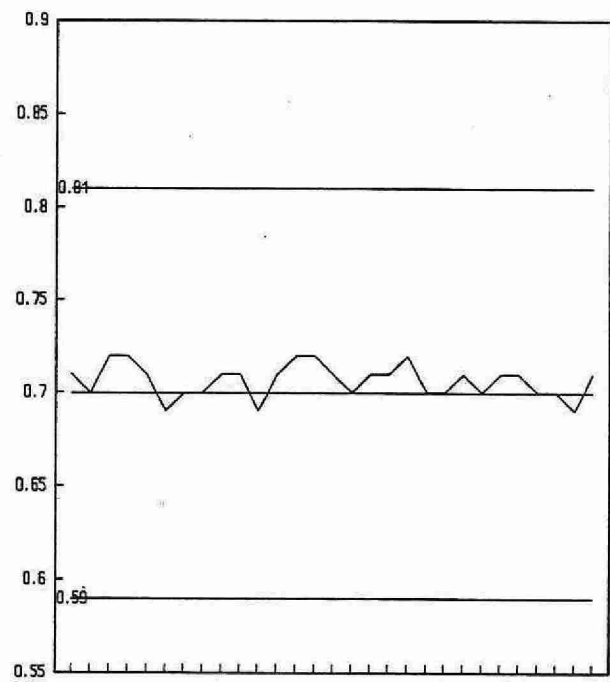
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 50              | 3.00 - 5.00                  | 0.0098                    | 0.2                            |
| 8               | 5.01 - 8.50                  | 0.0708                    | 1.3                            |
| 1               | 8.51 - 14.0                  | N.A.                      | N.A.                           |
| 59              | Overall                      | 0.0130                    |                                |

pH

QUALITY CONTROL DATA FROM 10/01/94 TO 14/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

pH

**IDENTIFICATION:**

|                     |                        |                   |               |
|---------------------|------------------------|-------------------|---------------|
| Laboratory Unit     | Titration              | Method Introduced | 09/07/80      |
| Method Reference No | E3289A                 | Units             | Dimensionless |
| LIMS Product Code   | PHALCO3289, CONDPH3289 | Supervisor        | F. Lo         |
| Sample Type/Matrix  | Rivers, Lakes          |                   |               |

**SAMPLING:**

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or Plastic |

**ANALYTICAL PROCEDURE:**

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Gran Alkalinity, total fixed endpoint alkalinity, and conductivity are determined simultaneously.

**INSTRUMENTATION:**

Automated modular titration system with microcomputer control and data processing software.

**REPORTING:**

Maximum Significant Figures: 3

**CALIBRATION:**

2 standard buffers covering the pH range of 4 to 9

**CONTROLS:**

|             |   |
|-------------|---|
| Calibration | 2 QC standards e.g. QCA   |
| Drift       | In run standards throughout the run (diluted tap water 20% V/V) |

# pH

QUALITY CONTROL DATA FROM 06/01/94 TO 12/12/94

Laboratory Unit: Titration

Analytical Range: to 14.00 Dimensionless

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 87 | 7.41                      | 7.413                 | 0.003     | 0.0245                    |
| B:   | 87 | 4.45                      | 4.490                 | 0.040     | 0.0322                    |
| A+B: | 87 | 11.86                     | 11.903                | 0.043     | 0.0385                    |
| A-B: | 87 | 2.96                      | 2.923                 | -0.037    | 0.0422                    |

s.d.(AB) S(between runs): 0.029

S/Sw:(within run): 0.030

S/Sw: 0.97

On any given day the calibration is accepted if the values obtained lie within the ranges:

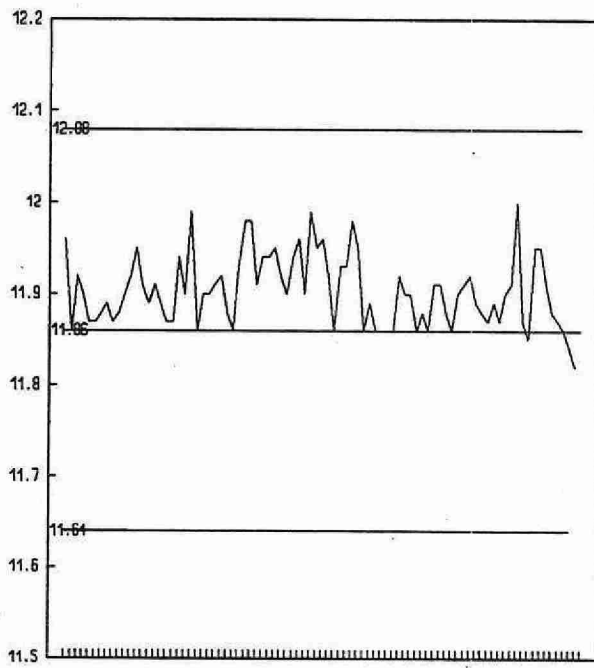
11.64 - 12.08 for A+B  
2.79 - 3.13 for A-B

## DUPLICATES:

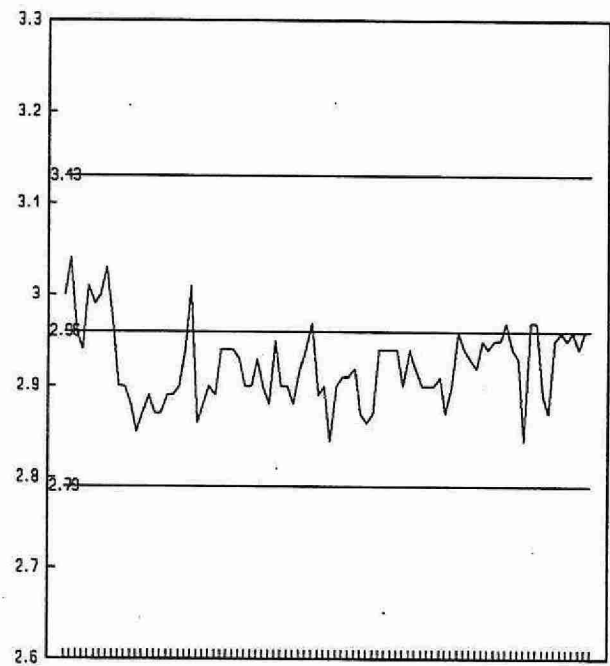
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 30              | 4.00 - 7.50                  | 0.0889                    | 1.3                            |
| 68              | 7.51 - 8.00                  | 0.0751                    | 0.9                            |
| 150             | 8.01 - 9.00                  | 0.0628                    | 0.7                            |
| 248             | Overall                      | 0.0691                    |                                |

pH

QUALITY CONTROL DATA FROM 06/01/94 TO 12/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## PHENOLICS, REACTIVE

### IDENTIFICATION:

|                     |   |                   |                |
|---------------------|---|-------------------|----------------|
| Laboratory Unit     | Colourimetry  | Method Introduced | 01/04/74       |
| Method Reference No | E3179A  | Units             | µg/L as Phenol |
| LIMS Product Code   | PHEN3179  | Supervisor        | M. Rawlings    |
| Sample Type/Matrix  | Rivers, Lakes, Precipitation, Soil Extracts, Effluents, Domestic Water Supplies, Leachates, Sewage, Industrial Wastes |                   |                |

### SAMPLING:

|                   |  |
|-------------------|--|
| Quantity Required | 250 mL   |
| Container         | Glass, (Phenol bottle with white cap containing preservative is available) |
| Preservative      | Sulfuric acid to pH 1.5 - 2  |

### ANALYTICAL PROCEDURE:

Samples are automatically distilled from an acid media, and reactive phenolics in the distillate are determined colourimetrically by formation of an antipyrene dye through reactions with 4-aminoantipyrene and potassium ferricyanide.

Approximate absorbance: 0.03 at the full scale level.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 505 nm.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

BL plus 2 standards

### CONTROLS:

|             |                                   |
|-------------|-----------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA   |
| Drift       | BL, standard, BL every 10 samples |

### NOTES:

A report identifying reactive phenolics is available on request.

# PHENOLICS, REACTIVE

QUALITY CONTROL DATA FROM 07/01/94 TO 23/12/94

Laboratory Unit: Colourimetry

Full Scale: to 50.0 µg/L as Phenol

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 74 | 40                        | 40.32                 | -0.322    | 0.5209                    |
| B:   | 74 | 10                        | 10.15                 | -0.146    | 0.1761                    |
| A+B: | 74 | 50                        | 50.47                 | -0.468    | 0.5354                    |
| A-B: | 74 | 30                        | 30.18                 | -0.176    | 0.5639                    |

s.d.(AB)

S(between runs): 0.39

Sw(within run): 0.40

S/Sw: 0.97

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

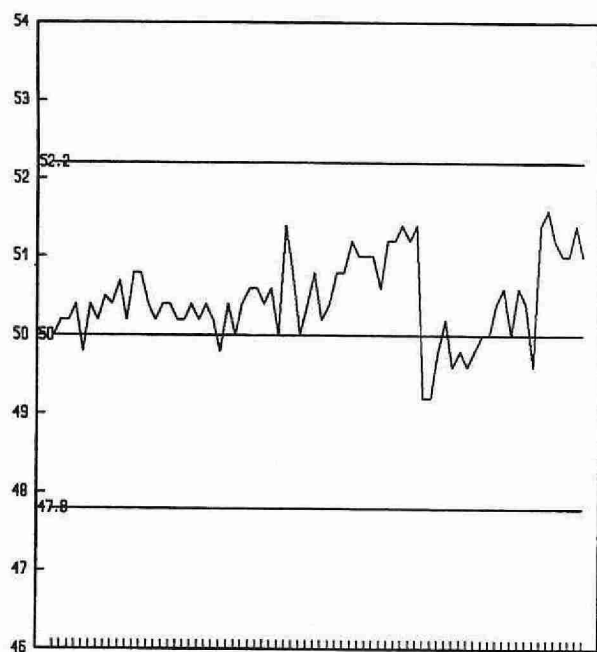
47.8 - 52.2 for A+B  
28.5 - 31.5 for A-B

## DUPLICATES:

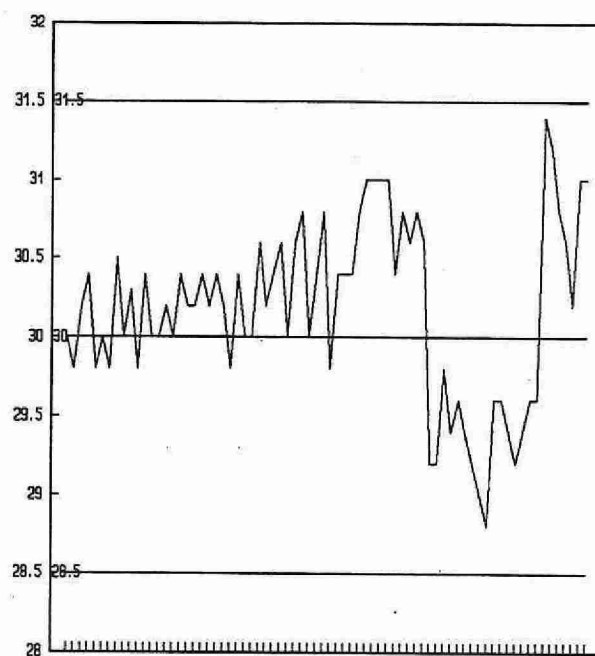
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 211             | 0.00 - 5.0                   | 0.1610                    | 28.5                           |
| 3               | 5.1 - 10.0                   | 0.4985                    | 6.1                            |
| 4               | 10.1 - 25.0                  | 0.2689                    | 1.5                            |
| 3               | 25.1 - 50.0                  | 1.1608                    | 3.1                            |
| 221             | Overall                      | 0.1672                    |                                |

**PHENOLICS, REACTIVE** ( $\mu\text{g/L}$  as Phenol)

QUALITY CONTROL DATA FROM 07/01/94 TO 23/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT



## PHOSPHORUS, REACTIVE ortho-PHOSPHATE

### IDENTIFICATION:

|                     |  |                   |             |
|---------------------|--|-------------------|-------------|
| Laboratory Unit     | Colourimetry   | Method Introduced | 01/04/79    |
| Method Reference No | E3364A   | Units             | mg/L as P   |
| LIMS Product Code   | DISNUT3364   | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Rivers, Lakes, Precipitation, Soil Extracts, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.2 at the full scale level.

Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                         |                         |
|--------------------------------|-------------------------|-------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.0005 | Current T value: 0.0025 |
|--------------------------------|-------------------------|-------------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g. QCA                |
| Drift       | BL every 10 samples; standard every 20 samples |

### NOTES:

Sept.'94 the method codes RNDNP-E3174A, E3175A, E3208A and E3266 were amalgamated and a new method code RNDNP-E3364A was generated.

# PHOSPHORUS, REACTIVE ortho-PHOSPHATE

QUALITY CONTROL DATA FROM 07/01/94 TO 04/12/94

Laboratory Unit: Colourimetry

Full Scale: to 0.100 mg/L as N

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 94 | 0.080                  | 0.0799             | -0.0001   | 0.0010                 |
| B:   | 94 | 0.040                  | 0.0397             | -0.0003   | 0.0008                 |
| C:   | 94 | 0.008                  | 0.0084             | 0.0004    | 0.0008                 |
| A+B: | 94 | 0.120                  | 0.1196             | -0.0004   | 0.0014                 |
| A-B: | 94 | 0.040                  | 0.0401             | 0.0001    | 0.0012                 |
| B+C: | 94 | 0.048                  | 0.0482             | 0.0002    | 0.0013                 |
| B-C: | 94 | 0.032                  | 0.0313             | -0.0007   | 0.0008                 |

s.d.(AB) S(between runs): 0.0009

Sw(within run): 0.0008

S/Sw: 1.1

s.d.(BC) S(between runs): 0.0008

Sw(within run): 0.0005

S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

|        |   |        |     |     |
|--------|---|--------|-----|-----|
| 0.1150 | - | 0.1250 | for | A+B |
| 0.0364 | - | 0.0436 | for | A-B |
| 0.0450 | - | 0.0510 | for | B+C |
| 0.0296 | - | 0.0344 | for | B-C |

## DUPLICATES:

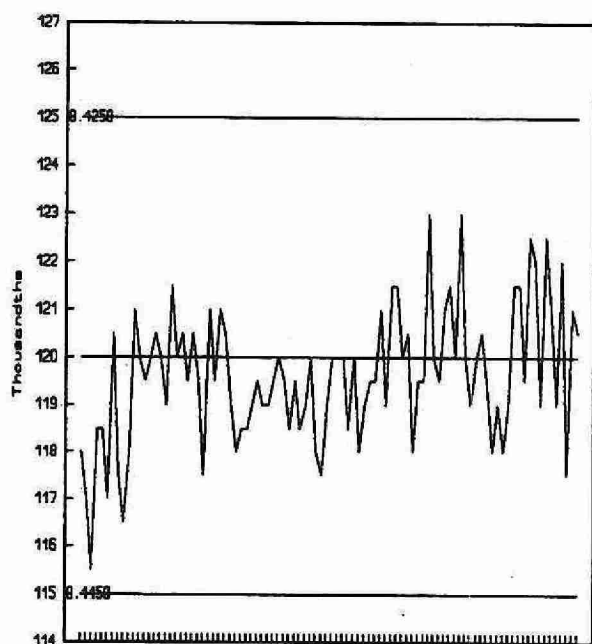
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 203          | 0.000 - 0.010             | 0.00078                | 58.2                        |
| 25           | 0.011 - 0.020             | 0.00134                | 15.1                        |
| 25           | 0.021 - 0.050             | 0.00172                | 4.9                         |
| 9            | 0.051 - 0.100             | 0.00209                | 2.9                         |
| 262          | Overall                   | 0.00095                |                             |

## OTHER CHECKS:

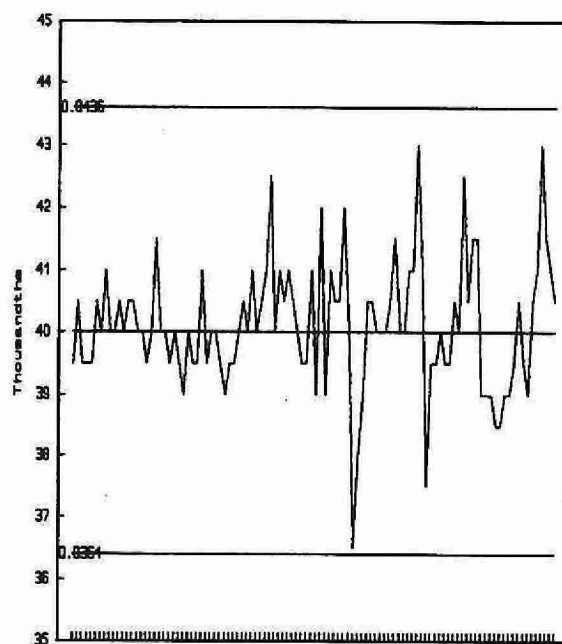
|                 | n  | Mean    | Standard Deviation (1) |
|-----------------|----|---------|------------------------|
| Long Term Blank | 94 | 0.00002 | 0.00080                |

PHOSPHORUS, REACTIVE ortho-PHOSPHATE (mg/L as P)

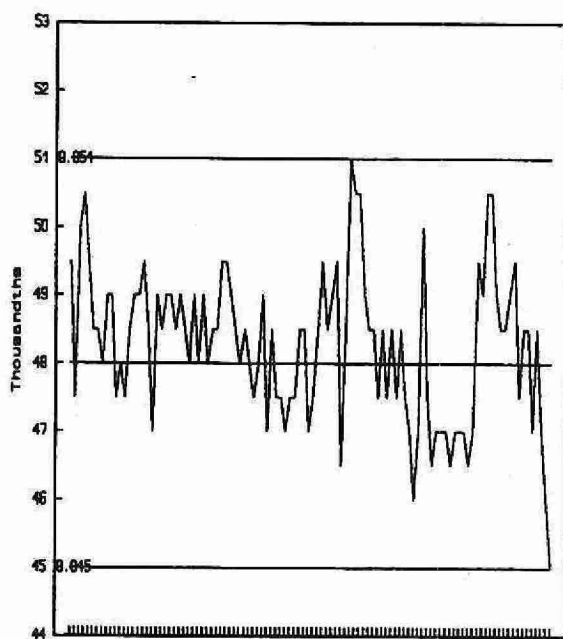
QUALITY CONTROL DATA FROM 07/01/94 TO 04/12/94



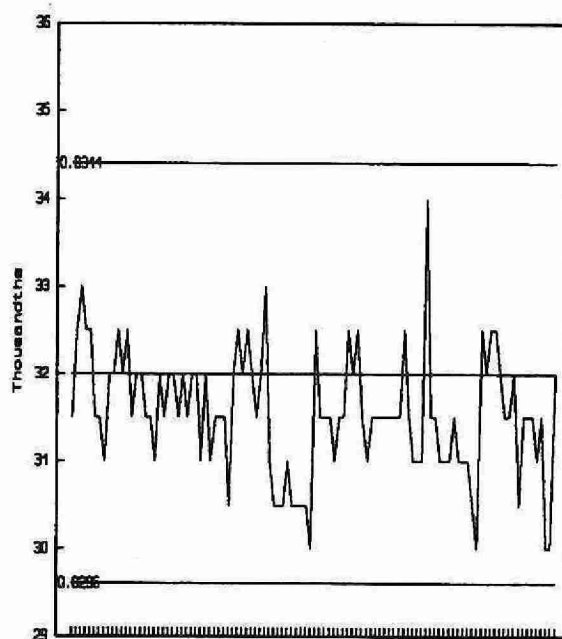
QUALITY CONTROL SAMPLE A+B



QUALITY CONTROL SAMPLE A-B



QUALITY CONTROL SAMPLE B+C



QUALITY CONTROL SAMPLE B-C

CONTROL LIMIT

## PHOSPHORUS, REACTIVE ortho-PHOSPHATE

### IDENTIFICATION:

|                     |  |                   |             |
|---------------------|--|-------------------|-------------|
| Laboratory Unit     | Colourimetry   | Method Introduced | 01/04/79    |
| Method Reference No | E3366A   | Units             | mg/L as P   |
| LIMS Product Code   | DISNUT3366   | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Sewage, Industrial Waste, Domestic Waters, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 10 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.5 at the full scale level.

Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                       |                      |
|--------------------------------|-----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.1 |
|--------------------------------|-----------------------|----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g. QCA                |
| Drift       | BL every 10 samples; standard every 20 samples |

### NOTES:

Sept.'94 the method codes SDNP-E3223A, E3193A, E3184A and E3185 were amalgamated and a new method code SDNP-E3366A was generated.

# PHOSPHORUS, REACTIVE ortho-PHOSPHATE

QUALITY CONTROL DATA FROM 07/01/94 TO 21/12/94

Laboratory Unit: Colourimetry

Full Scale: to 10.0 mg/L as P

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 83 | 8.00                   | 8.003              | 0.003     | 0.0452                 |
| B:   | 83 | 4.00                   | 4.001              | 0.001     | 0.0261                 |
| C:   | 83 | 0.800                  | 0.804              | 0.004     | 0.0125                 |
| A+B: | 83 | 12.0                   | 12.003             | 0.003     | 0.0579                 |
| A-B: | 83 | 4.00                   | 4.003              | 0.003     | 0.0457                 |
| B+C: | 83 | 4.80                   | 4.805              | 0.005     | 0.0308                 |
| B-C: | 83 | 3.20                   | 3.196              | -0.004    | 0.0269                 |

s.d.(AB) S(between runs): 0.037

Sw(within run): 0.032

S/Sw: 1.1

s.d.(BC) S(between runs): 0.021

Sw(within run): 0.019

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 11.7 | - | 12.3 | for | A+B |
| 3.78 | - | 4.22 | for | A-B |
| 4.66 | - | 4.94 | for | B+C |
| 3.09 | - | 3.31 | for | B-C |

## DUPLICATES:

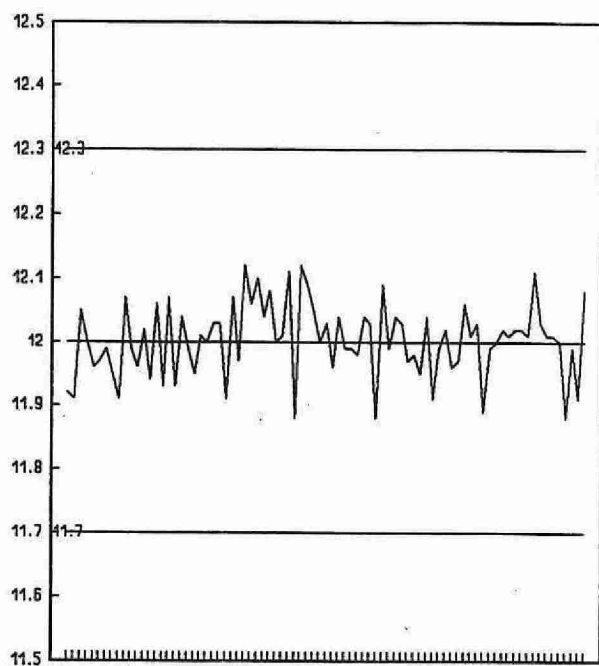
| n Data Pairs | Sample Concentration Span. | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|----------------------------|------------------------|-----------------------------|
| 192          | 0.00 - 1.00                | 0.0120                 | 16.4                        |
| 18           | 1.01 - 2.00                | 0.0413                 | 3.2                         |
| 26           | 2.01 - 5.00                | 0.1241                 | 4.4                         |
| 5            | 5.01 - 10.0                | 0.0654                 | 1.1                         |
| 241          | Overall                    | 0.0180                 |                             |

## OTHER CHECKS:

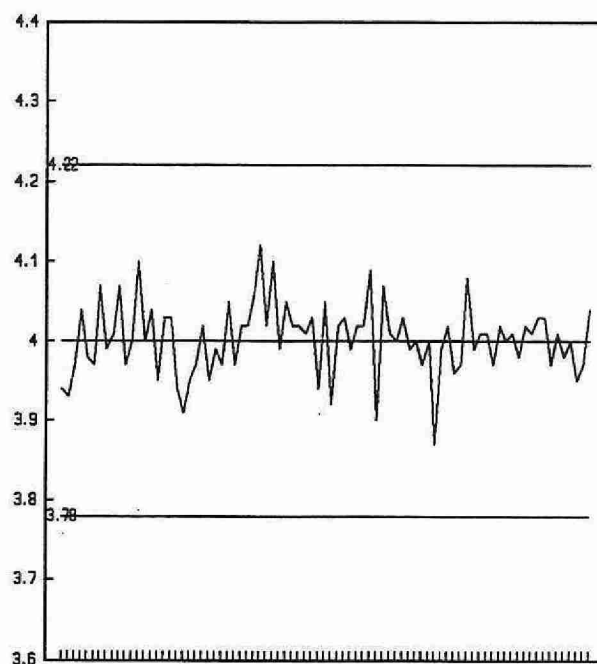
|                 | n  | Mean     | Standard Deviation (1) |
|-----------------|----|----------|------------------------|
| Long Term Blank | 83 | -0.00012 | 0.00065                |

PHOSPHORUS, REACTIVE ortho-PHOSPHATE (mg/L as P)

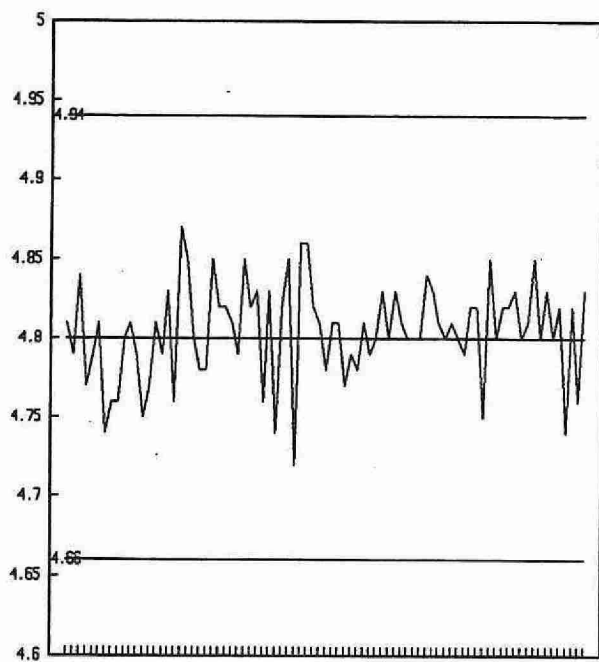
QUALITY CONTROL DATA FROM 06/01/93 TO 20/12/93



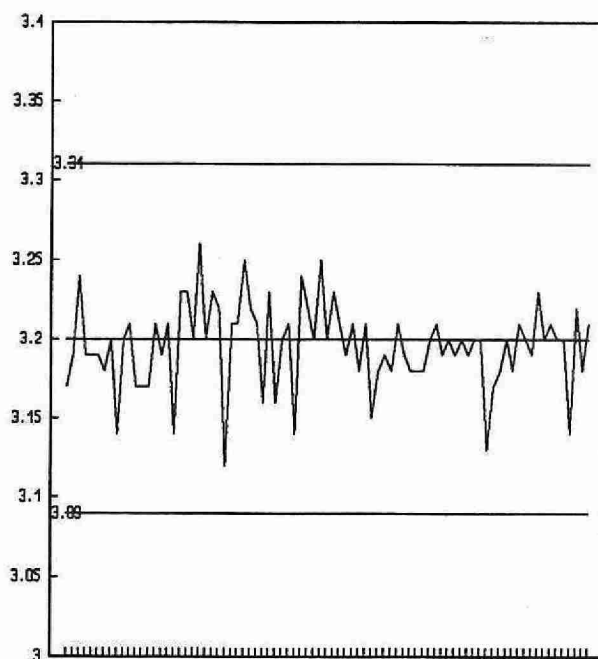
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B-C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## PHOSPHORUS, TOTAL

### IDENTIFICATION:

|                      |                               |                   |            |
|----------------------|-------------------------------|-------------------|------------|
| Laboratory Unit      | Dorset                        | Method Introduced | 22/03/79   |
| LIS Test Name Code   | PPUT1,PPUT2                   | Units             | µg/L as P  |
| Work Station Code    | DOP                           | Unit Code         | 063815     |
| Method Code          | 5926C2                        | Supervisor        | J. McBride |
| Method Reference No. | E3036A                        |                   |            |
| Sample Type/Matrix:  | Streams, Lakes, Precipitation |                   |            |

### SAMPLING:

|                    |   |
|--------------------|---|
| Quantity Required: | 35 mL   |
| Container:         | Specially marked Pyrex culture tubes with Teflon-lined caps |

### ANALYTICAL PROCEDURE:

After withdrawal of excess volume, digestion reagent is added and samples are autoclaved in sulphuric acid-potassium persulphate media at 121°C for 60 min. The orthophosphate content of the digestate is determined colourimetrically by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.3 at the full scale level

### INSTRUMENTATION:

Autoclave plus basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube. Two analytical ranges are obtained from the output of the colourimeter.

### REPORTING:

|                                |                      |                    |
|--------------------------------|----------------------|--------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1 |
|--------------------------------|----------------------|--------------------|

### CALIBRATION:

BL plus 9 undigested standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 undigested standards, e.g. QCA                             |
| Drift       | BL every 10 samples and BL plus 1 undigested standard every 20 samples |
| Recovery    | 3 digested BL plus 3 digested standards, e.g. R1                       |

### NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.



# PHOSPHORUS, TOTAL

QUALITY CONTROL DATA FROM 13/01/94 TO 23/12/94

Laboratory: Dorset

Full Scale: to 100.0 µg/L as P

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 81 | 45.0                   | 44.98              | -0.02     | 1.1238                 |
| B:   | 81 | 13.5                   | 14.06              | 0.56      | 0.5116                 |
| C:   | 81 | 4.5                    | 4.54               | 0.04      | 0.3494                 |
| A+B: | 81 | 58.5                   | 59.03              | 0.53      | 1.1120                 |
| A-B: | 81 | 31.5                   | 30.92              | -0.58     | 1.3461                 |
| B+C: | 81 | 18.0                   | 18.59              | 0.59      | 0.6780                 |
| B-C: | 81 | 9.0                    | 9.52               | 0.52      | 0.5561                 |

s.d.(AB) S(between runs): 0.87 Sw(within run): 0.95 S/Sw: 0.92  
s.d.(BC) S(between runs): 0.44 Sw(within run): 0.39 S/Sw: 1.11

The calibration is accepted if the calibration control values obtained lie within the ranges:

63.5 - 53.5 for A+B  
35.5 - 27.5 for A-B  
20 - 16 for B+C  
10.5 - 7.5 for B-C

## RECOVERIES:

| Number of Data | Expected Concentration | Mean Concentration | Standard Deviation (1) |
|----------------|------------------------|--------------------|------------------------|
| 81             | 34.5                   | 34.72              | 1.0066                 |
| 81             | 13.5                   | 14.02              | 0.7983                 |
| 81             | 6.6                    | 7.01               | 0.7489                 |

## DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 119          | 0.00 - 10.0               | 0.2063                 | 5.6                         |
| 67           | 10.1 - 20.0               | 0.3378                 | 3.9                         |
| 38           | 20.1 - 50.0               | 0.4220                 | 1.7                         |
| 6            | 50.1 - 100.0              | 0.7672                 | 1.0                         |
| 230          | Overall                   | 0.2884                 |                             |

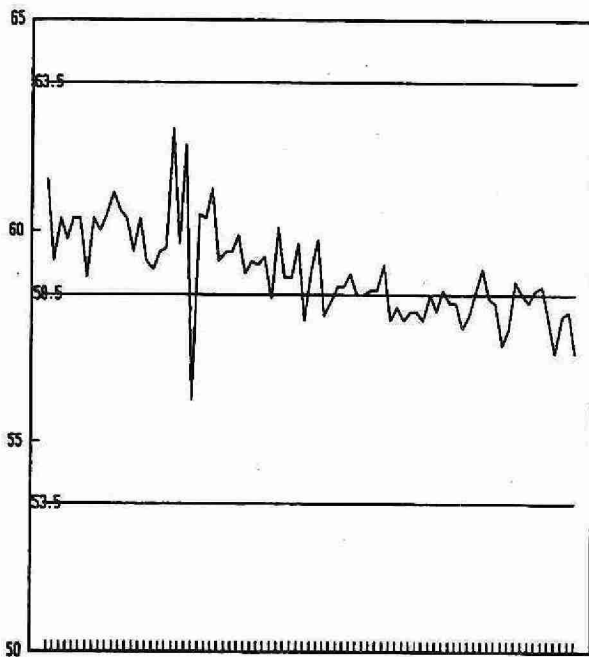
## OTHER CHECKS:

|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 81 | -0.04 | 0.2300                 |
| Digested Blank  | 81 | 1.04  | 0.4321                 |

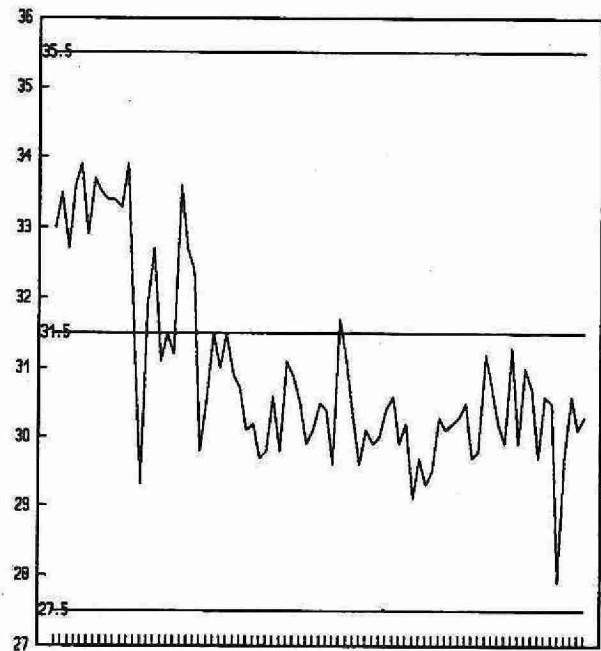


**PHOSPHORUS, TOTAL** ( $\mu\text{g/L}$  as P)

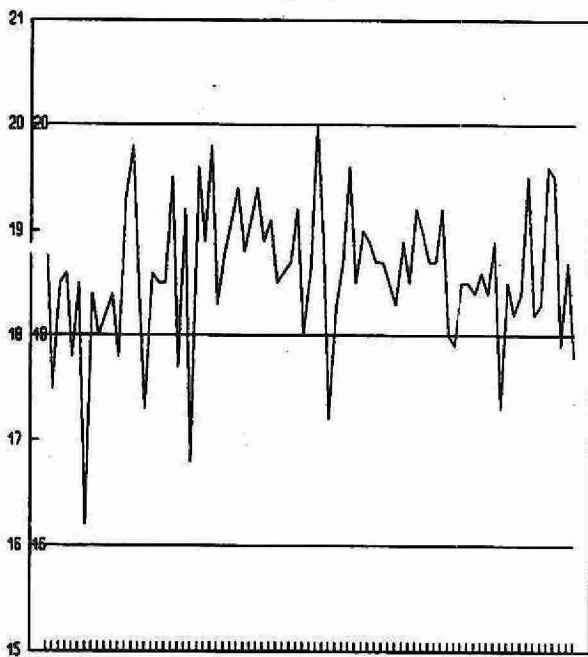
QUALITY CONTROL DATA FROM 13/01/94 TO 23/12/94



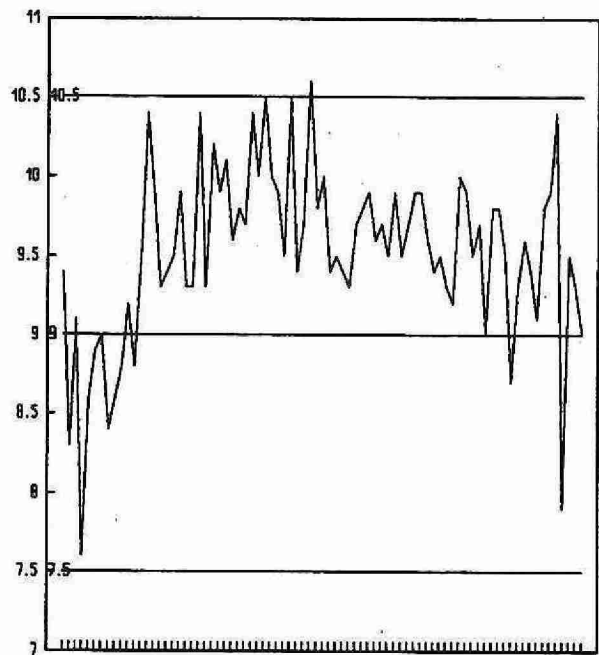
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## PHOSPHORUS, TOTAL

### IDENTIFICATION:

|                     |                   |                   |             |
|---------------------|-------------------|-------------------|-------------|
| Laboratory Unit     | Colourimetry      | Method Introduced | Mar '89     |
| Method Reference No | E3116A            | Units             | mg/g as P   |
| LIMS Product Code   | TNP3116           | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Soil and Sediment |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 0.08 to 0.4 g    |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Phosphorus compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

### INSTRUMENTATION:

Hot plate

Basic automated modular continuous flow system : Colourimetric measurement is through a 5 cm. light path at 660 nm.

Data capture, and processing via a microcomputer system

### REPORTING:

#### SEDIMENT/SOIL

|   |                               |                               |
|---|-------------------------------|-------------------------------|
| Maximum Significant Figures: 2 decimal places | Current W value:<br>0.01/0.02 | Current T value:<br>0.05/0.10 |
|---|-------------------------------|-------------------------------|

### CALIBRATION:

3 High and 2 Low Calibration Standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | In house composite B-Soil/sediment, plus QC Soils/Sediment (RS92)       |
| Drift       | 4 BL's per run; high and low calibration standard at the end of the run |
| Recovery    | 1 digested BL plus 4 digested standards                                 |

### NOTES:

System is calibrated with undigested standards.

# PHOSPHORUS, TOTAL

QUALITY CONTROL DATA FROM 07/01/94 TO 14/06/94

Laboratory Unit: Colourimetry

Full Scale: 0 to 5 mg/g as P

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| RS92 | 68 | 0.47                      | 0.468                 | -0.018    | 0.0236                    |

The calibration is accepted if the calibration control values obtained lie within the ranges:  
0.40 - 0.54 for RS92

## DUPLICATES: (Sediment)

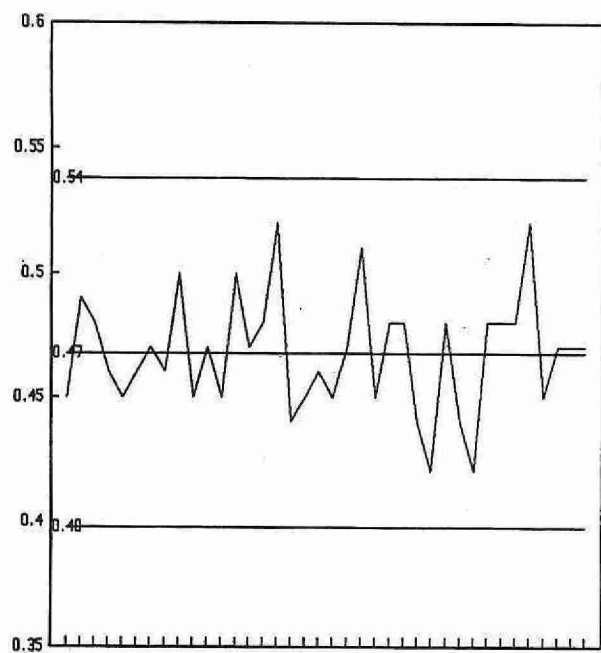
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 19              | 0.00 - 0.50                  | 0.0291                    | 7.1                            |
| 28              | 0.51 - 1.00                  | 0.0401                    | 4.5                            |
| 18              | 1.01 - 2.50                  | 0.0660                    | 4.5                            |
| 3               | 2.51 - 5.00                  | 0.2195                    | 5.0                            |
| 68              | Overall                      | 0.0471                    |                                |

## DUPLICATES: (Soils)

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 5               | 0.00 - 0.50                  | 0.0280                    | 5.4                            |
| 31              | 5.01 - 1.00                  | 0.0502                    | 5.6                            |
| 37              | 1.01 - 2.50                  | 0.0650                    | 4.7                            |
| 0               | 2.51 - 5.00                  | N.A.                      | N.A.                           |
| 73              | Overall                      | 0.0549                    |                                |

PHOSPHORUS, TOTAL (mg/g as P)

QUALITY CONTROL DATA FROM 07/01/94 TO 14/07/94



QUALITY CONTROL STANDARD  
RS92 - Sediment and soil control

\_\_\_\_\_ CONTROL LIMIT

## PHOSPHORUS, TOTAL

### IDENTIFICATION:

|                     |                                    |                   |             |
|---------------------|------------------------------------|-------------------|-------------|
| Laboratory Unit     | Colourimetry                       | Method Introduced | Mar '89     |
| Method Reference No | E3118A                             | Units             | mg/g as P   |
| LIMS Product Code   | TNP3118                            | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Terrestrial and aquatic vegetation |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 0.02 to 0.04 g   |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Phosphorus compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

### INSTRUMENTATION:

Hot plate

Basic automated modular continuous flow system : Colourimetric measurement is through a 5 cm. light path at 660 nm.

Data capture, and processing via a microcomputer system

### REPORTING:

|   |                       |                       |
|---|-----------------------|-----------------------|
| Maximum Significant Figures: 2 decimal places | Current W value: 0.02 | Current T value: 0.10 |
|---|-----------------------|-----------------------|

### CALIBRATION:

3 High and 2 Low Calibration Standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | In house composite A-VEG, plus QC VEG (Pine Needles)                    |
| Drift       | 4 BL's per run; high and low calibration standard at the end of the run |
| Recovery    | 1 digested BL plus 4 digested standards                                 |

### NOTES:

System is calibrated with undigested standards.

## PHOSPHORUS, TOTAL

QUALITY CONTROL DATA FROM 05/04/94 TO 17/10/94

Laboratory Unit: Colourimetry

Full Scale: 0 to 8 mg/g as P

### CALIBRATION CONTROL:

|                                 | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|---------------------------------|----|---------------------------|-----------------------|-----------|---------------------------|
| Pine Needles<br>(non certified) | 20 | 1.20                      | 1.23                  | 0.03      | 0.0415                    |

The calibration is accepted if the calibration control values obtained lie within the ranges:

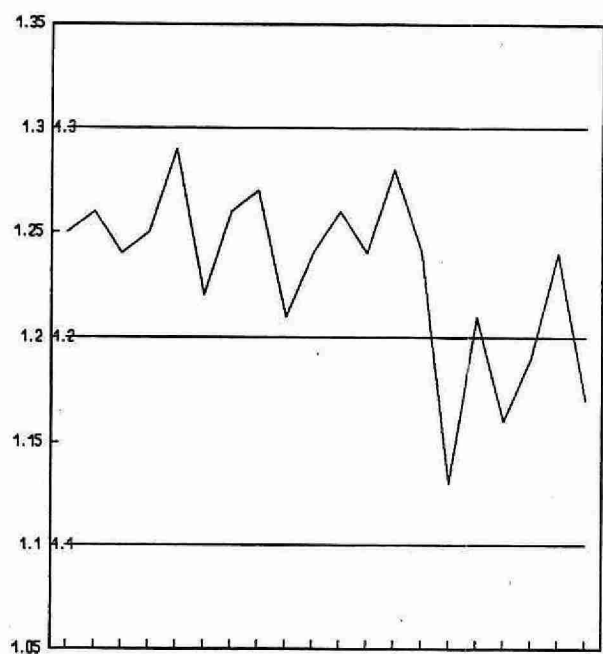
1.1 - 1.3 for Pine Needles

### DUPLICATES: (Vegetation)

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 0               | 0.00 - 0.80                  | N.A.                      | N.A.                           |
| 10              | 0.81 - 1.60                  | 0.0510                    | 3.3                            |
| 41              | 1.61 - 4.00                  | 0.1220                    | 4.5                            |
| 3               | 4.01 - 8.00                  | 0.5293                    | 8.8                            |
| 54              | Overall                      | 0.1229                    |                                |

PHOSPHORUS, TOTAL (mg/g as P)

QUALITY CONTROL DATA FROM 05/04/94 TO 17/10/94



QUALITY CONTROL STANDARD  
Pine Needle

CONTROL LIMIT

## PHOSPHORUS, TOTAL

### IDENTIFICATION:

|                     |  |                   |             |
|---------------------|--|-------------------|-------------|
| Laboratory Unit     | Colourimetry   | Method Introduced | 01/04/79    |
| Method Reference No | E3367A   | Units             | mg/L as P   |
| LIMS Product Code   | TOTNUT3367   | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Rivers, Lakes, Precipitation, Soil Extracts, Effluents |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digesters kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.4 at the full scale level.

Total Kjeldahl nitrogen is determined simultaneously.

### INSTRUMENTATION:

Three Block digesters

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system

### REPORTING:

|                                |                        |                       |
|--------------------------------|------------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.002 | Current T value: 0.01 |
|--------------------------------|------------------------|-----------------------|

### CALIBRATION:

BL plus 7 undigested standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 undigested standards, e.g. QCA                    |
| Drift       | BL every 10 samples; undigested standard every 20 samples     |
| Recovery    | 3 digested BL plus 3 digested standards in duplicate, e.g. R1 |



# PHOSPHORUS, TOTAL

QUALITY CONTROL DATA FROM 06/01/94 TO 28/12/94

Laboratory Unit: Colourimetry

Full Scale: to 0.200 mg/L as P

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 135 | 0.160                  | 0.161              | 0.001     | 0.0011                 |
| B:   | 135 | 0.080                  | 0.0802             | 0.0002    | 0.0006                 |
| C:   | 135 | 0.016                  | 0.0157             | -0.003    | 0.0008                 |
| A+B: | 135 | 0.240                  | 0.241              | 0.001     | 0.0012                 |
| A-B: | 135 | 0.080                  | 0.081              | 0.001     | 0.0014                 |
| B+C: | 135 | 0.096                  | 0.0959             | -0.0001   | 0.0011                 |
| B-C: | 135 | 0.064                  | 0.0644             | 0.004     | 0.0009                 |

s.d.(AB) S(between runs): 0.0009 Sw(within run): 0.0010 S/Sw: 0.96  
s.d.(BC) S(between runs): 0.0007 Sw(within run): 0.0006 S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.233 - 0.247 for A+B  
0.075 - 0.085 for A-B  
0.092 - 0.100 for B+C  
0.061 - 0.067 for B-C

## RECOVERIES:

| Number of Data | Expected Concentration | Mean Concentration | Standard Deviation (1) |
|----------------|------------------------|--------------------|------------------------|
| 135            | 0.140                  | 0.1370             | 0.0047                 |
| 135            | 0.084                  | 0.0822             | 0.0034                 |
| 135            | 0.028                  | 0.0278             | 0.0029                 |

## DUPLICATES:

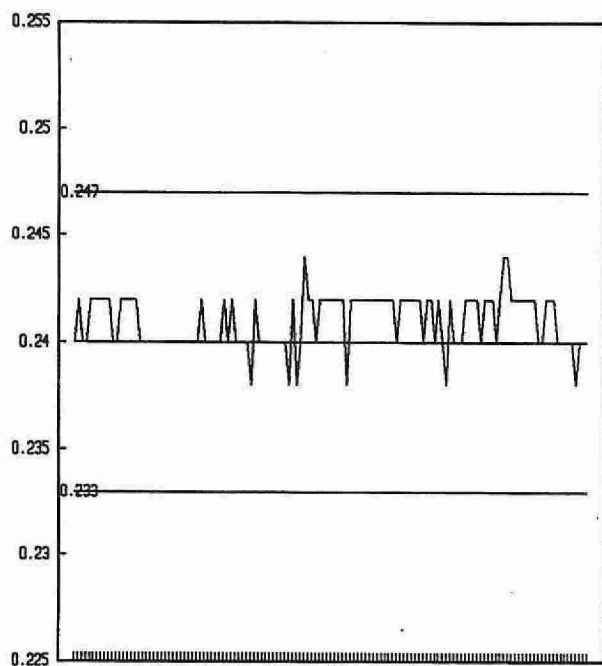
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 262          | 0.000 - 0.020             | 0.0017                 | 47.7                        |
| 60           | 0.021 - 0.040             | 0.0030                 | 44.5                        |
| 43           | 0.041 - 0.100             | 0.0030                 | 14.5                        |
| 24           | 0.101 - 0.200             | 0.0046                 | 3.9                         |
| 389          | Overall                   | 0.0021                 |                             |

## OTHER CHECKS:

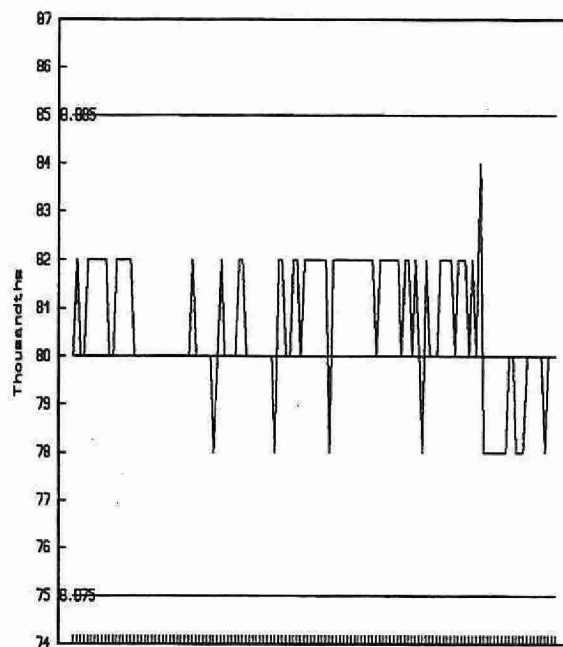
|                 | n   | Mean    | Standard Deviation (1) |
|-----------------|-----|---------|------------------------|
| Long Term Blank | 135 | 0.00013 | 0.00056                |
| Digested Blank  | 135 | 0.0029  | 0.0033                 |

# PHOSPHORUS, TOTAL (mg/L as P)

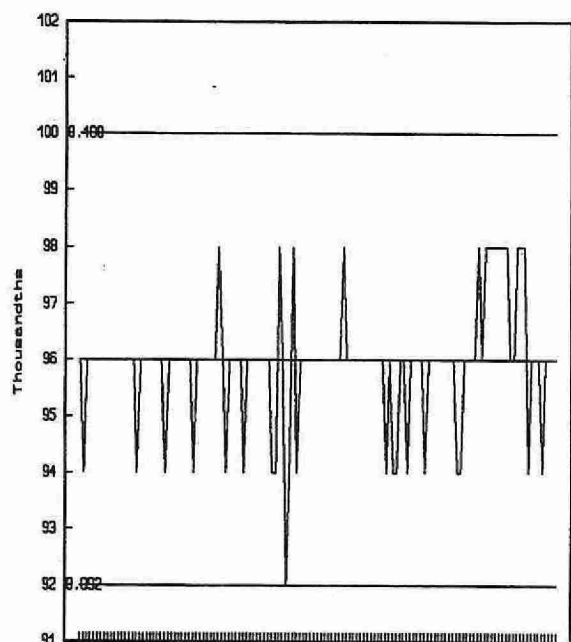
QUALITY CONTROL DATA FROM 06/01/94 TO 28/12/94



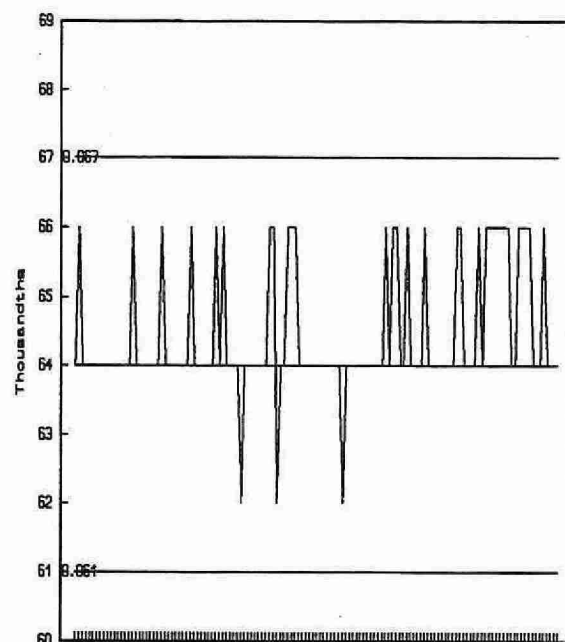
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## PHOSPHORUS, TOTAL

### IDENTIFICATION:

|                     |   |                   |             |
|---------------------|---|-------------------|-------------|
| Laboratory Unit     | Colourimetry  | Method Introduced | 01/04/79    |
| Method Reference No | E3368A  | Units             | mg/L as P   |
| LIMS Product Code   | TOTNUT3368  | Supervisor        | M. Rawlings |
| Sample Type/Matrix  | Sewage, Industrial Waste, Domestic Waters, Effluents, Leachates |                   |             |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.8 at the full scale level.

Total Kjeldahl Nitrogen is determined simultaneously.

### INSTRUMENTATION:

3-Block digesters

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using an IR sensitive phototube. Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.10 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 standards, e.g. QCA                               |
| Drift       | BL every 10 samples; undigested standard every 20 samples     |
| Recovery    | 3 digested BL plus 3 digested standards in duplicate, e.g. R1 |

### NOTES:

System is calibrated with undigested standards.

# PHOSPHORUS, TOTAL

QUALITY CONTROL DATA FROM 05/01/94 TO 20/12/94

Laboratory Unit: Colourimetry

Full Scale: to 10.0 mg/L as P

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 84 | 8.0                    | 7.993              | 0.0069    | 0.0243                 |
| B:   | 84 | 4.0                    | 3.997              | 0.0033    | 0.0175                 |
| C:   | 84 | 0.8                    | 0.807              | -0.0069   | 0.0144                 |
| A+B: | 84 | 12.0                   | 11.990             | 0.0102    | 0.0339                 |
| A-B: | 84 | 4.0                    | 3.996              | 0.0036    | 0.0254                 |
| B+C: | 84 | 4.8                    | 4.804              | -0.0036   | 0.0278                 |
| B-C: | 84 | 3.2                    | 3.190              | 0.0102    | 0.0160                 |

s.d.(AB)

S(between runs):

0.0212

Sw(within run):

0.0180

S/Sw: 1.2

s.d.(BC)

S(between runs):

0.0160

Sw(within run):

0.0113

S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 11.9 | - | 12.1 | for | A+B |
| 3.9  | - | 4.1  | for | A-B |
| 4.7  | - | 4.9  | for | B+C |
| 3.15 | - | 3.25 | for | B-C |

## RECOVERIES:

| Number of Data | Expected Concentration | Mean Concentration | Standard Deviation (1) |
|----------------|------------------------|--------------------|------------------------|
| 84             | 7                      | 6.914              | 0.1141                 |
| 84             | 4.2                    | 4.152              | 0.0656                 |
| 84             | 1.4                    | 1.385              | 0.0404                 |

## DUPLICATES:

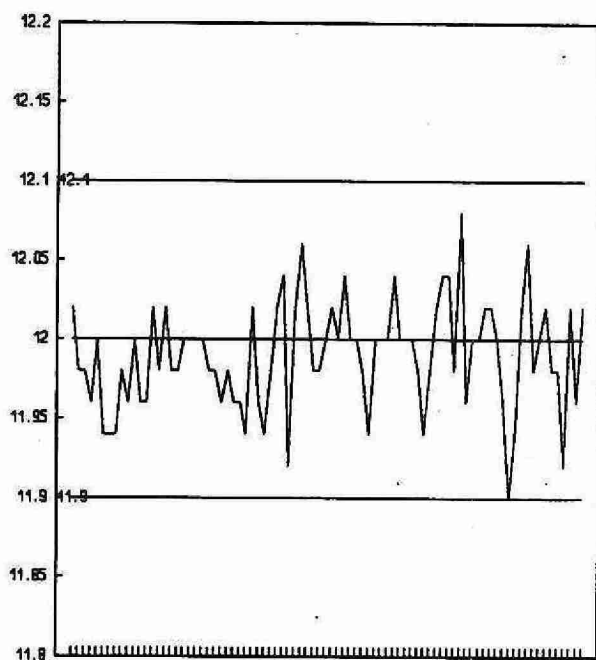
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 149          | 0.00 - 1.00               | 0.0142                 | 20.9                        |
| 13           | 1.00 - 2.00               | 0.0505                 | 2.9                         |
| 61           | 2.00 - 5.00               | 0.1146                 | 3.2                         |
| 26           | 5.00 - 10.00              | 0.1447                 | 2.0                         |
| 249          | Overall                   | 0.0464                 |                             |

## OTHER CHECKS:

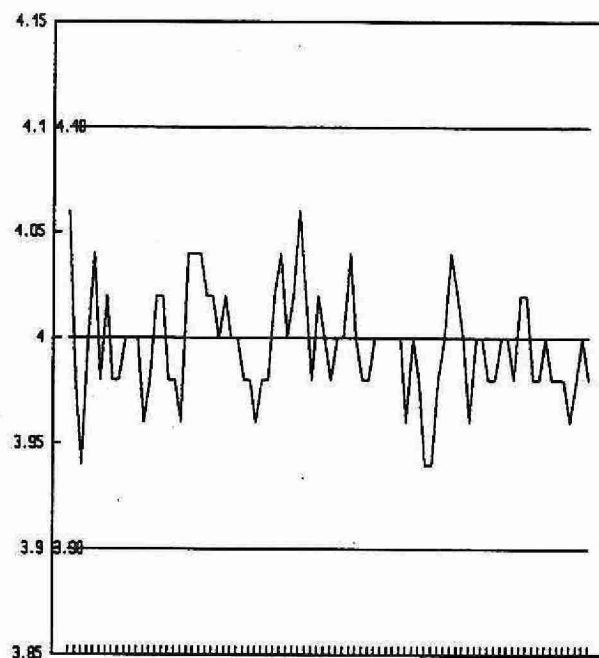
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 84 | 0.005 | 0.0107                 |
| Digested Blank  | 84 | 0.011 | 0.0149                 |

**PHOSPHORUS, TOTAL** (mg/L as P)

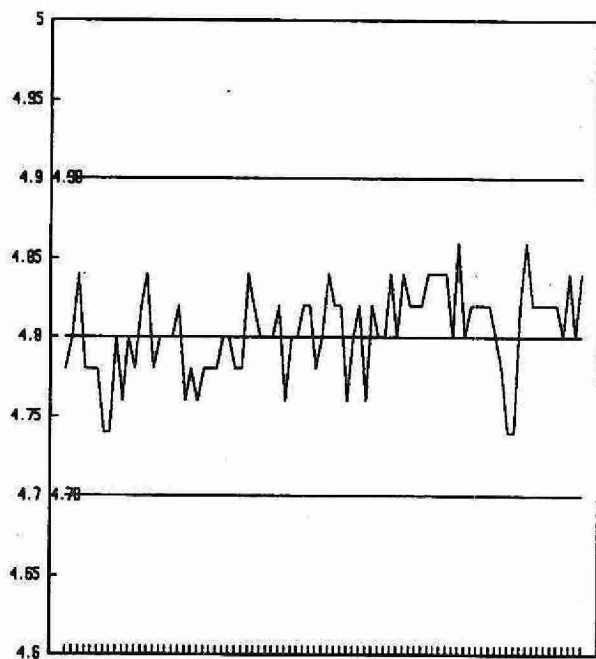
QUALITY CONTROL DATA FROM 05/01/94 TO 20/12/94



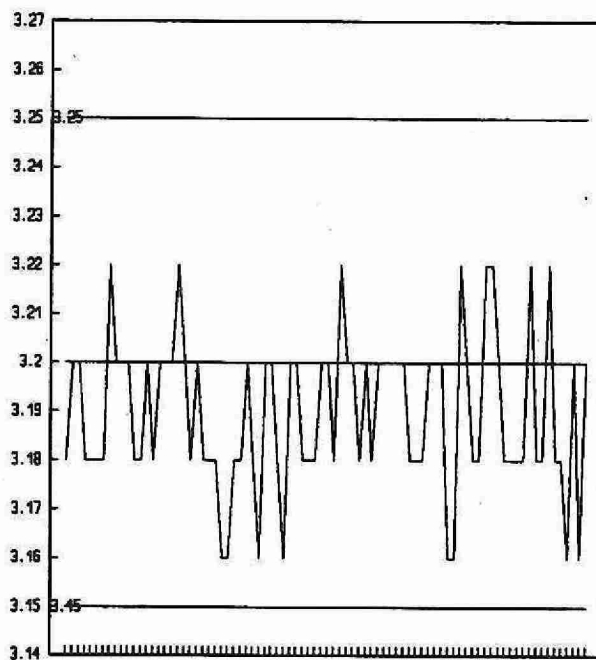
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## POTASSIUM

### IDENTIFICATION:

|                      |  |                   |  |
|----------------------|--|-------------------|--|
| Laboratory Unit      | Atomic Absorption                      | Method Introduced | 18/05/79                                 |
| Method Reference No. | E3146A                                 | Units             | mg/L as K, ( $\mu\text{g}$ /filter as K) |
| LIMS Product Code    | CAT3146, (NAK3146)                     | Supervisor        | J. McBride                               |
| Sample Type/Matrix   | Precipitation, (LOVOL Filter Extracts) |                   |  |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 5 mL    |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

### REPORTING:

| Maximum Significant Figures | Current W value |                             | Current T value |                             |
|-----------------------------|-----------------|-----------------------------|-----------------|-----------------------------|
| 3                           | 0.002 mg/L      | (0.1 $\mu\text{g}$ /filter) | 0.010 mg/L      | (0.5 $\mu\text{g}$ /filter) |

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 2 standards, e.g., QCA       |
| Drift       | BL, reslope standard every 10 samples. |

# POTASSIUM

QUALITY CONTROL DATA FROM 11/01/94 TO 15/12/94

Laboratory Unit: Atomic Absorption

Full Scale: to 1.00 mg/L as K

## CALIBRATION CONTROL:

|      | n  | Expected<br>Concentration | Mean<br>Concentration | Mean Bias | Standard<br>Deviation (1) |
|------|----|---------------------------|-----------------------|-----------|---------------------------|
| A:   | 25 | 0.60                      | 0.6065                | 0.0065    | 0.0067                    |
| B:   | 25 | 0.10                      | 0.1018                | 0.0018    | 0.0030                    |
| A+B: | 25 | 0.70                      | 0.7083                | 0.0083    | 0.0081                    |
| A-B: | 25 | 0.50                      | 0.5048                | 0.0048    | 0.0065                    |

s.d.(AB)      S(between runs): 0.0052      Sw(within run): 0.0046      S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.672 - 0.728 for A+B  
0.479 - 0.521 for A-B

## DUPLICATES:

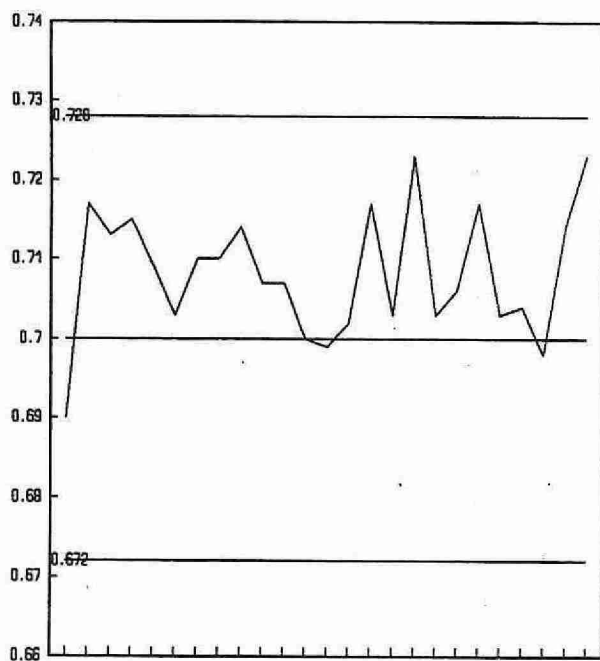
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 41              | 0.000 - 0.100                | 0.0011                    | 3.6                            |
| 7               | 0.101 - 0.200                | 0.0062                    | 7.0                            |
| 2               | 0.201 - 0.500                | N.A.                      | N.A.                           |
| 7               | 0.501 - 1.000                | 0.0039                    | 0.7                            |
| 57              | Overall                      | 0.0017                    |                                |

## OTHER CHECKS:

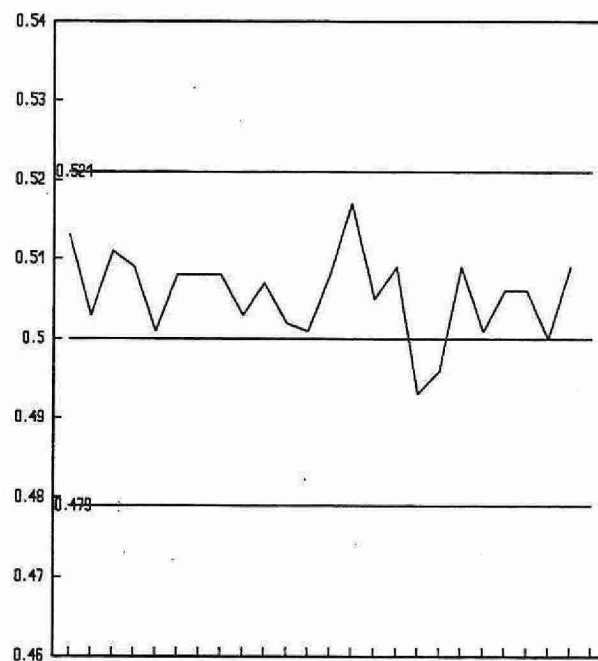
|                 | n  | Mean   | Standard<br>Deviation (1) |
|-----------------|----|--------|---------------------------|
| Long Term Blank | 25 | 0.0002 | 0.0039                    |

**POTASSIUM** (mg/L as K)

QUALITY CONTROL DATA FROM 11/01/94 TO 15/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

\_\_\_\_\_  
CONTROL LIMIT



## POTASSIUM

### IDENTIFICATION:

|                      |                                      |                   |            |
|----------------------|--------------------------------------|-------------------|------------|
| Laboratory Unit      | Atomic Absorption                    | Method Introduced | 01/04/74   |
| Method Reference No. | E3171A                               | Units             | mg/L as K  |
| LIMS Product Code    | CAT3171,NAK3171                      | Supervisor        | J. McBride |
| Sample Type/Matrix   | Surface Waters, DWSP Drinking Waters |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.923 at the full scale value.

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.01 | Current T value: 0.05 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 11 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA                   |
| Drift       | BL every 10 samples; 2 standards every 20 samples. |

### NOTES:

Control limits were exceeded on Aug 15,22 and Nov 18, 1994.

# POTASSIUM

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94

Laboratory Unit: Atomic Absorption

Full Scale: to 5.00 mg/L as K

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 154 | 4.00                   | 4.01               | 0.01      | 0.0416                 |
| B:   | 154 | 1.00                   | 1.002              | 0.002     | 0.0194                 |
| C:   | 154 | 0.25                   | 0.253              | 0.003     | 0.0068                 |
| A+B: | 154 | 5.00                   | 5.02               | 0.02      | 0.0515                 |
| A-B: | 154 | 3.00                   | 3.01               | 0.01      | 0.0395                 |
| B+C: | 154 | 1.25                   | 1.26               | 0.01      | 0.0227                 |
| B-C: | 154 | 0.75                   | 0.749              | -0.001    | 0.0181                 |

s.d.(AB) S(between runs): 0.032

Sw(within run): 0.027

S/Sw: 1.2

s.d.(BC) S(between runs): 0.015

Sw(within run): 0.013

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 4.77  | - | 5.23  | for | A+B |
| 2.85  | - | 3.15  | for | A-B |
| 1.175 | - | 1.325 | for | B+C |
| 0.700 | - | 0.800 | for | B-C |

## DUPLICATES:

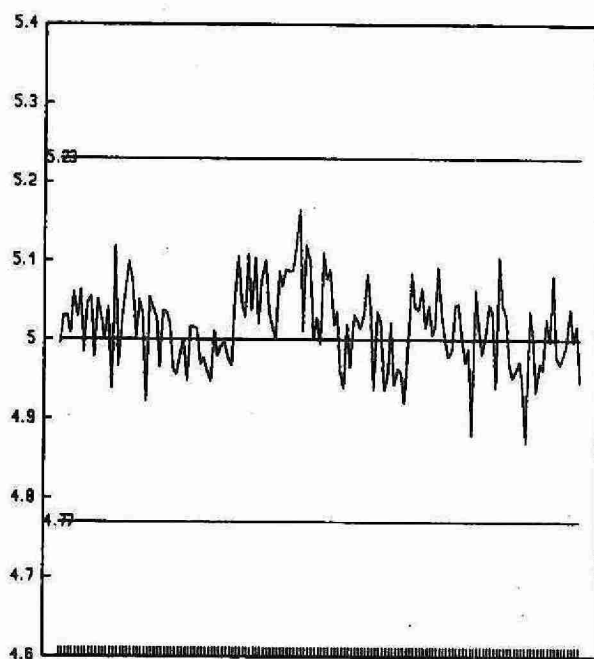
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 16           | 0.000 - 0.500             | 0.0062                 | 2.3                         |
| 49           | 0.501 - 1.00              | 0.0086                 | 1.5                         |
| 166          | 1.01 - 2.50               | 0.0184                 | 1.9                         |
| 53           | 2.51 - 5.00               | 0.0444                 | 1.7                         |
| 284          | Overall                   | 0.0193                 |                             |

## OTHER CHECKS:

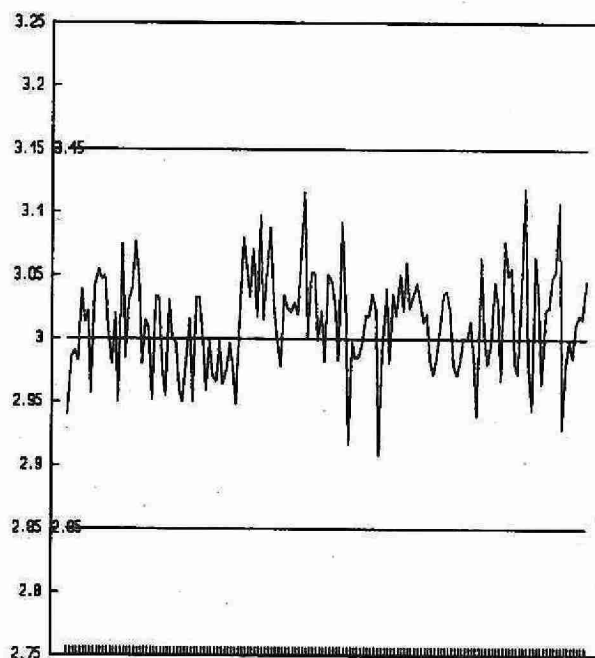
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 154 | 0.0003 | 0.0057                 |

# POTASSIUM (mg/L as K)

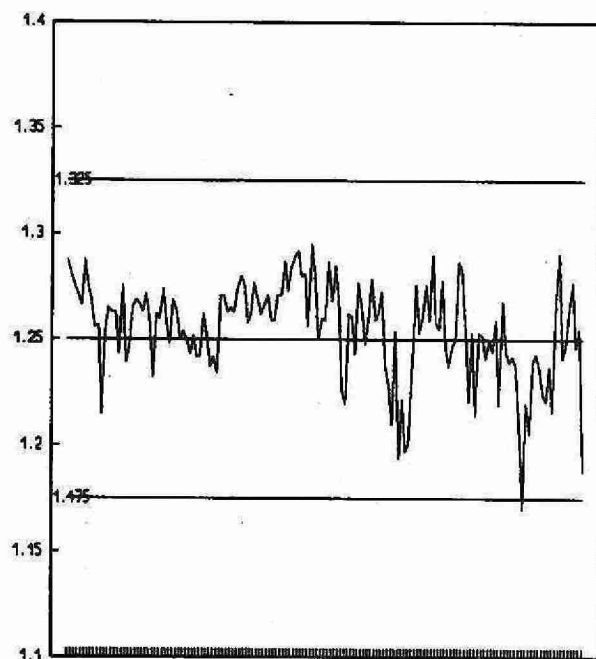
QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94



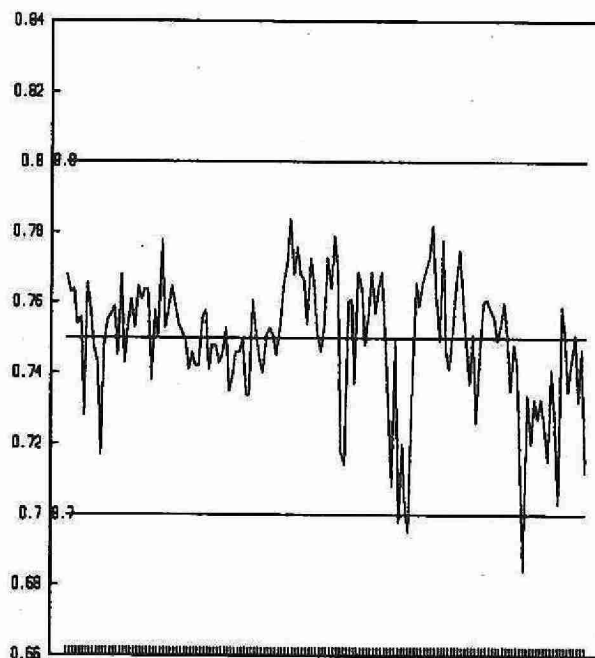
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## POTASSIUM

### IDENTIFICATION:

|                      |  |                   |            |
|----------------------|--|-------------------|------------|
| Laboratory Unit      | Atomic Absorption  | Method Introduced | 08/04/86   |
| Method Reference No. | E3217A   | Units             | mg/L as K  |
| LIMS Product Code    | CAT3217,K3217  | Supervisor        | J. McBride |
| Sample Type/Matrix   | Domestic Waters, Leachates, Effluents, Sewage, Industrial Wastes |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 1.16 at full scale level.

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 11 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA                   |
| Drift       | BL every 10 samples; 2 standards every 20 samples. |

# POTASSIUM

QUALITY CONTROL DATA FROM 04/01/94 TO 29/12/94

Laboratory Unit: Absorption

Full Scale: to 25.0 mg/L as K

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 97 | 20.00                  | 19.98              | -0.02     | 0.2813                 |
| B:   | 97 | 5.00                   | 5.01               | 0.01      | 0.1052                 |
| C:   | 97 | 1.25                   | 1.26               | 0.01      | 0.0436                 |
| A+B: | 97 | 25.00                  | 24.99              | -0.01     | 0.3216                 |
| A-B: | 97 | 15.00                  | 14.97              | -0.03     | 0.2774                 |
| B+C: | 97 | 6.25                   | 6.27               | 0.02      | 0.1322                 |
| B-C: | 97 | 3.75                   | 3.749              | -0.001    | 0.0920                 |

s.d.(AB) S(between runs): 0.21

Sw(within run): 0.20

S/Sw: 1.1

s.d.(BC) S(between runs): 0.08

Sw(within run): 0.07

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 23.8 | - | 26.2 | for | A+B |
| 14.2 | - | 15.8 | for | A-B |
| 5.65 | - | 6.85 | for | B+C |
| 3.35 | - | 4.15 | for | B-C |

## DUPLICATES:

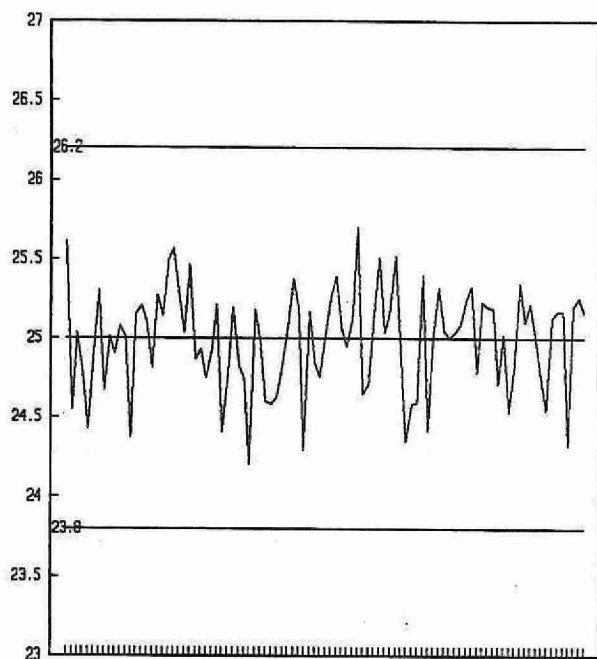
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 152          | 0.00 - 2.50               | 0.0390                 | 7.5                         |
| 34           | 2.51 - 5.00               | 0.0707                 | 2.2                         |
| 24           | 5.01 - 12.50              | 0.1444                 | 1.9                         |
| 9            | 12.51 - 25.00             | 0.2183                 | 1.5                         |
| 219          | Overall                   | 0.0567                 |                             |

## OTHER CHECKS:

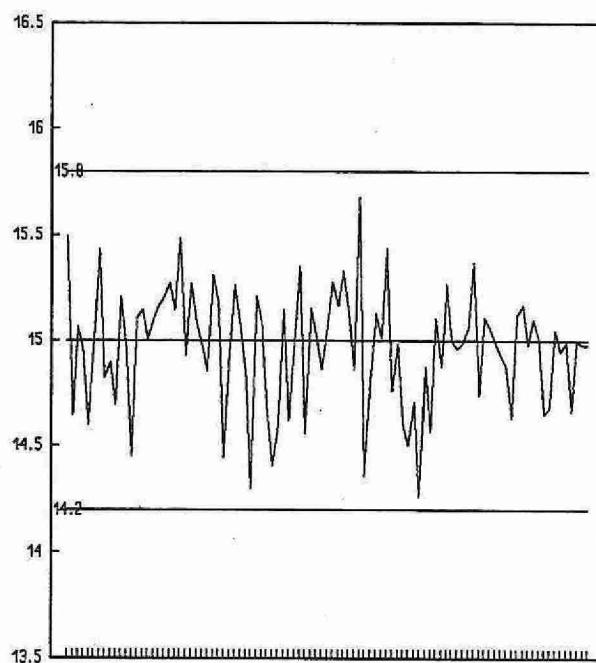
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 92 | -0.049 | 0.1000                 |

# POTASSIUM (mg/L as K)

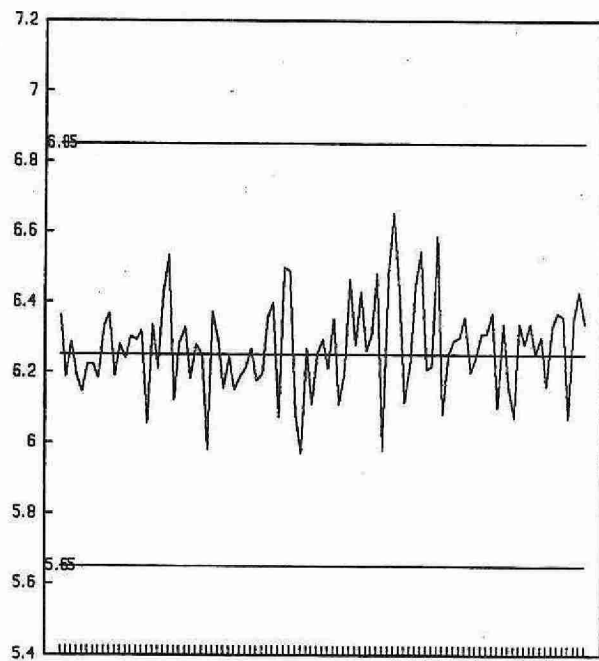
QUALITY CONTROL DATA FROM 04/01/94 TO 29/12/94



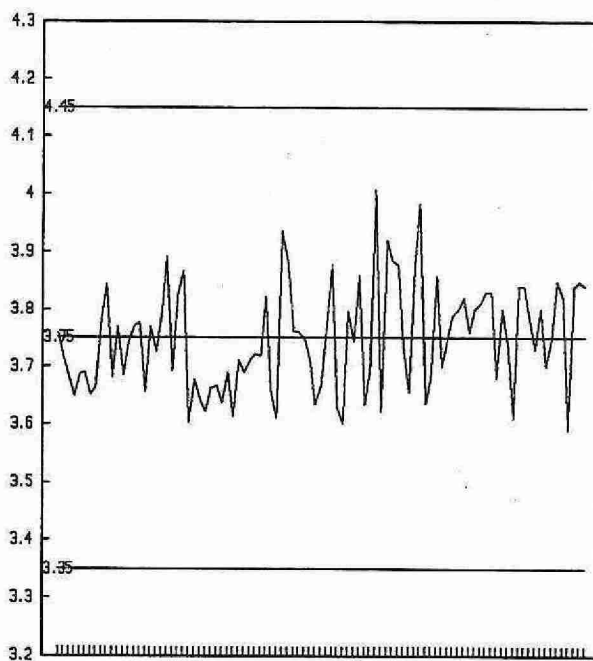
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## POTASSIUM

### IDENTIFICATION:

|                      |                |                   |            |
|----------------------|----------------|-------------------|------------|
| Laboratory Unit      | Dorset         | Method Introduced | 20/07/88   |
| LIS Test Name Code   | KKUR           | Units             | mg/L as K  |
| Work Station Code    | DOFLAME        | Unit Code         | 064819     |
| Method Code          | 002EA1         | Supervisor        | J. McBride |
| Method Reference No. | E3249A         |                   |            |
| Sample Type/Matrix   | Rivers, Lakes, |                   |            |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 5 mL    |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.005 | Current T value: 0.025 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA       |
| Drift       | BL, reslope standard every 10 samples. |

### NOTES:

The control standards are corrected for the LTB from which they were made.

# POTASSIUM

QUALITY CONTROL DATA FROM 17/01/94 TO 22/12/94

Laboratory Unit: Dorset

Full Scale: to 1.0 mg/L as K

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 74 | 0.80                   | 0.798              | -0.002    | 0.0081                 |
| B:   | 74 | 0.20                   | 0.200              | 0.000     | 0.0041                 |
| C:   | 74 | 0.05                   | 0.049              | -0.001    | 0.0032                 |
| A+B: | 74 | 1.00                   | 0.999              | -0.001    | 0.0105                 |
| A-B: | 74 | 0.60                   | 0.599              | -0.001    | 0.0082                 |
| B+C: | 74 | 0.25                   | 0.250              | 0.000     | 0.0062                 |
| B-C: | 74 | 0.15                   | 0.151              | 0.001     | 0.0044                 |

s.d.(AB) S(between runs): 0.006

Sw(within run): 0.006

S/Sw: 1.1

s.d.(BC) S(between runs): 0.004

Sw(within run): 0.003

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 0.973 | - | 1.027 | for | A+B |
| 0.580 | - | 0.620 | for | A-B |
| 0.228 | - | 0.272 | for | B+C |
| 0.134 | - | 0.166 | for | B-C |

## DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 49           | 0.00 - 0.10               | 0.0025                 | 12.5                        |
| 26           | 0.11 - 0.20               | 0.0088                 | 6.1                         |
| 79           | 0.21 - 0.50               | 0.0134                 | 5.7                         |
| 19           | 0.51 - 1.00               | 0.0202                 | 3.3                         |
| 173          | Overall                   | 0.0093                 |                             |

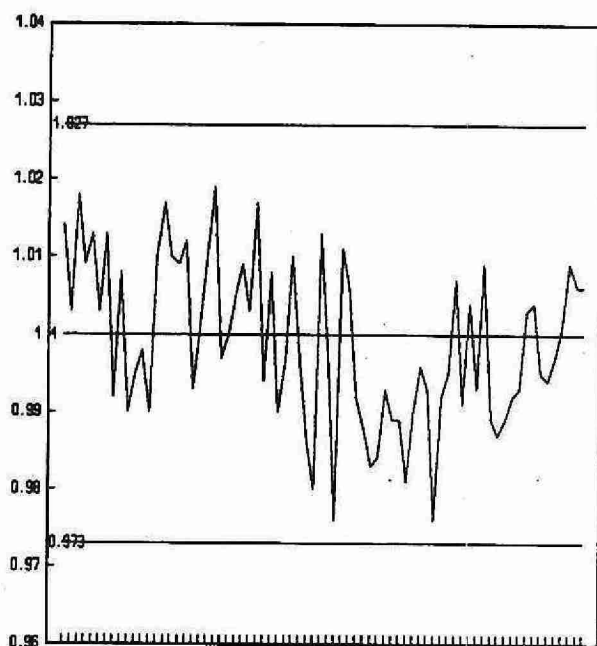
## OTHER CHECKS:

|                 | n  | Mean     | Standard Deviation (1) |
|-----------------|----|----------|------------------------|
| Long Term Blank | 74 | -0.00038 | 0.0027                 |

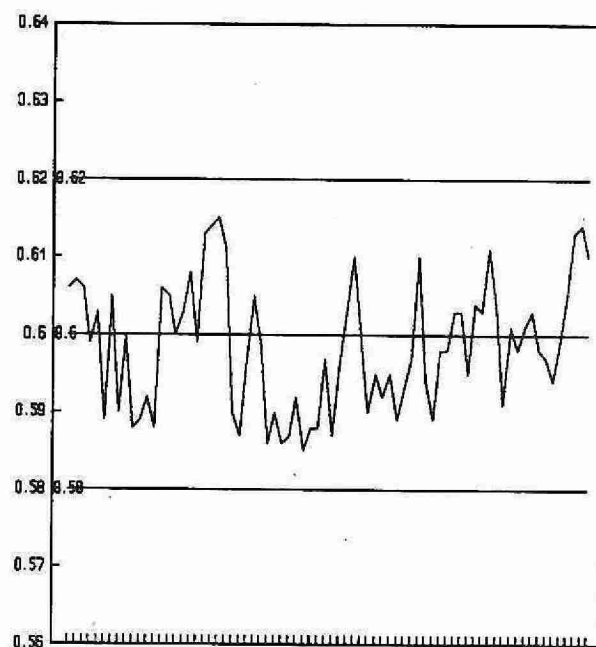


# POTASSIUM (mg/L as K)

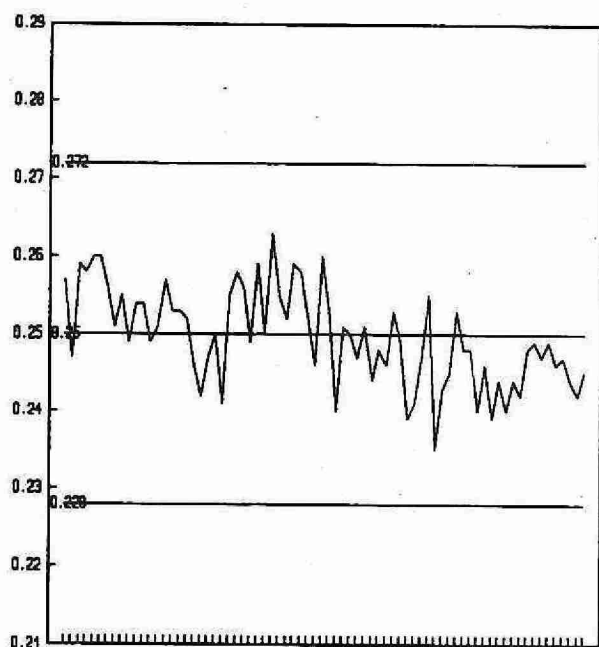
QUALITY CONTROL DATA FROM 17/01/94 TO 22/12/94



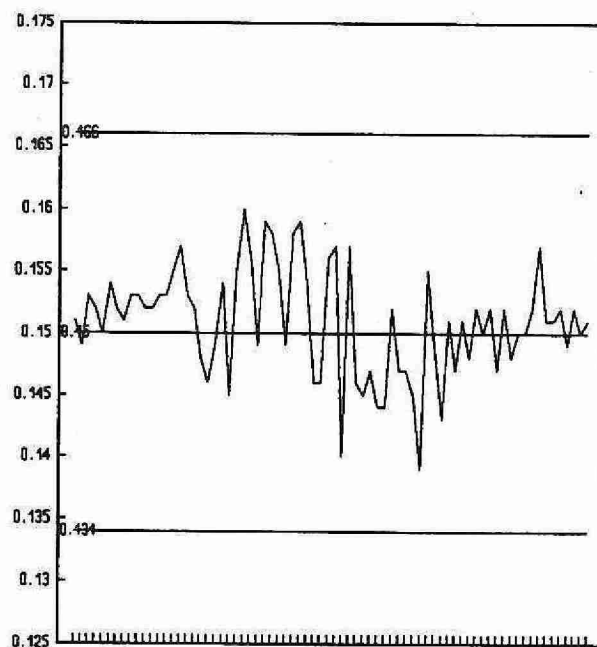
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## SILICON, REACTIVE SILICATES

### IDENTIFICATION:

|                      |  |                   |             |
|----------------------|--|-------------------|-------------|
| Laboratory Unit      | Colourimetry   | Method Introduced | 01/02/75    |
| Method Reference No. | E3370A   | Units             | mg/L as Si  |
| LIMS Product Code    | DCSI3370   | Supervisor        | M. Rawlings |
| Sample Type/Matrix   | Rivers, Lakes, Precipitation, Soil Extracts, Effluents, Domestic Water Supplies, Leachates |                   |             |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 10 mL   |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Reactive silicates are determined by formation of a reduced molybdo-silicate complex at pH 1.6, using ascorbic acid as the reducing agent, and oxalic acid to suppress phosphate interference.

Approximate absorbance: 0.7 at the full scale level.

Dissolved inorganic and dissolved organic carbon are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 660 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.10 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | LTBL plus 3 standards, e.g., QCA                |
| Drift       | BL every 10 samples; standard every 20 samples. |

### NOTES:

Sept.'94 the method codes ROM-E3176A, and E3178A were amalgamated and a new method code ROM-E3370A was generated.

# SILICON, REACTIVE SILICATES

QUALITY CONTROL DATA FROM 05/01/94 TO 22/10/94

Laboratory Unit: Colourimetry

Full Scale: to 10.0 mg/L as Si

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 146 | 8.00                   | 8.030              | 0.030     | 0.0379                 |
| B:   | 146 | 2.00                   | 2.000              | 0.000     | 0.0146                 |
| C:   | 146 | 0.50                   | 0.501              | 0.001     | 0.0049                 |
| A+B: | 146 | 10.00                  | 10.030             | 0.030     | 0.0488                 |
| A-B: | 146 | 6.00                   | 6.030              | 0.030     | 0.0304                 |
| B+C: | 146 | 2.50                   | 2.501              | 0.001     | 0.0168                 |
| B-C: | 146 | 1.50                   | 1.499              | 0.001     | 0.0139                 |

s.d.(AB) S(between runs): 0.029

Sw(within run): 0.022

S/Sw: 1.3

s.d.(BC) S(between runs): 0.011

Sw(within run): 0.010

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 9.70 | - | 10.3 | for | A+B |
| 5.80 | - | 6.20 | for | A-B |
| 2.30 | - | 2.70 | for | B+C |
| 1.38 | - | 1.62 | for | B-C |

## DUPLICATES:

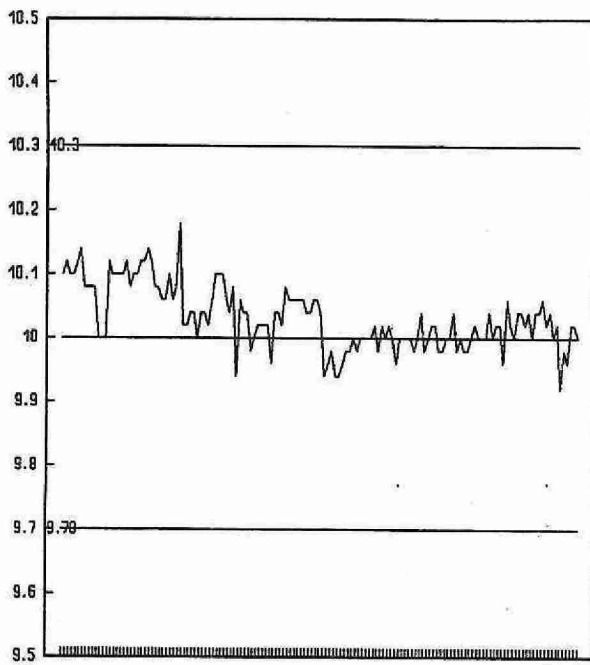
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 166          | 0.00 - 1.00               | 0.0299                 | 7.1                         |
| 67           | 1.01 - 2.00               | 0.0306                 | 2.0                         |
| 130          | 2.01 - 5.00               | 0.0193                 | 7.2                         |
| 64           | 5.01 - 10.0               | 0.1932                 | 2.8                         |
| 427          | Overall                   | 0.1547                 |                             |

## OTHER CHECKS:

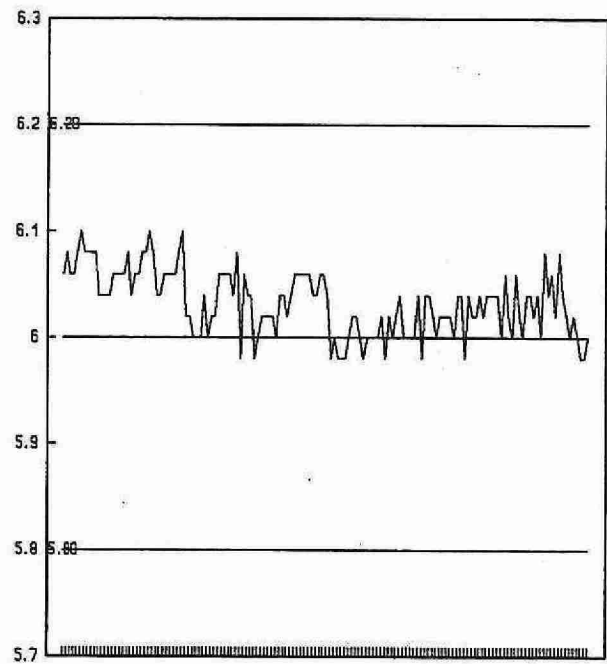
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 146 | 0.0008 | 0.0052                 |

SILICON, REACTIVE SILICATES (mg/L as Si)

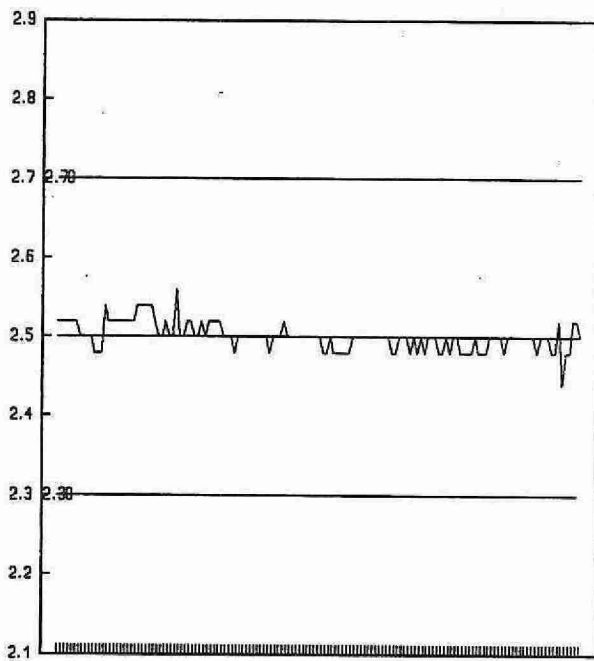
QUALITY CONTROL DATA FROM 05/01/94 TO 22/12/94



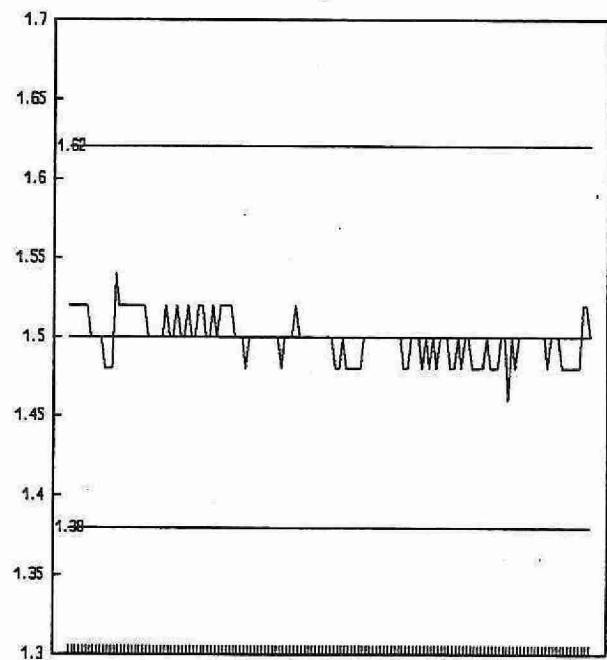
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

# SODIUM

## IDENTIFICATION:

|                      |  |                   |                         |
|----------------------|--|-------------------|-------------------------|
| Laboratory Unit      | Atomic Absorption                      | Method Introduced | 18/05/79                |
| Method Reference No. | E3146A                                 | Units             | mg/L as Na, (µg/Filter) |
| LIMS Product Code    | CAT3146, (NAK3146)                     | Supervisor        | J. McBride              |
| Sample Type/Matrix   | Precipitation, (LOVOL Filter Extracts) |                   |                         |

## SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 5 mL    |
| Container         | Plastic |

## ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

## INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

## REPORTING:

| Maximum Significant Figures | Current W value |               | Current T value |               |
|-----------------------------|-----------------|---------------|-----------------|---------------|
| 3                           | 0.002 mg/L      | 0.1 µg/Filter | 0.010 mg/L      | 0.5 µg/Filter |

## CALIBRATION:

BL plus 5 standards

## CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 2 standards, e.g., QCA       |
| Drift       | BL, reslope standard every 10 samples. |

# SODIUM

QUALITY CONTROL DATA FROM 11/01/94 TO 12/12/94

Laboratory Unit: Atomic Absorption

Full Scale: to 1.0 mg/L as Na

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 35 | 0.60                   | 0.6058             | 0.0058    | 0.0058                 |
| B:   | 35 | 0.10                   | 0.1047             | 0.0047    | 0.0044                 |
| A+B: | 35 | 0.70                   | 0.7105             | 0.0105    | 0.0078                 |
| A-B: | 35 | 0.50                   | 0.5010             | 0.0010    | 0.0066                 |

s.d.(AB)      S(between runs): 0.0051      Sw(within run): 0.0047      S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.671 - 0.729 for A+B  
0.478 - 0.522 for A-B

## DUPLICATES:

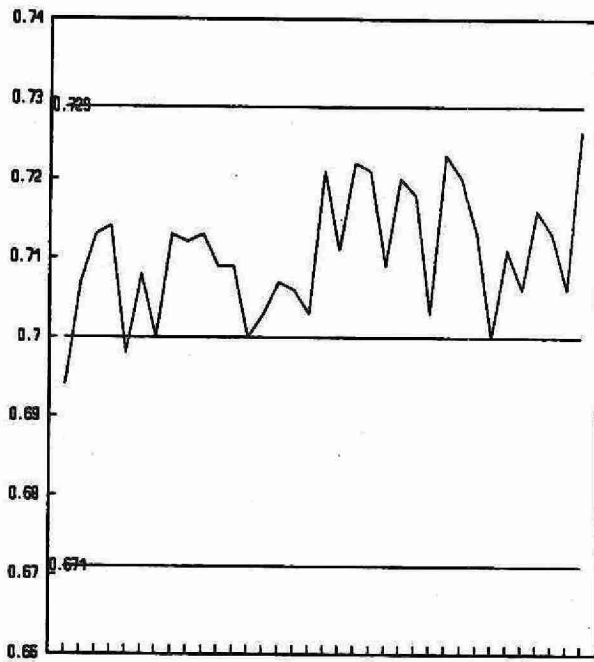
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 53           | 0.000 - 0.100             | 0.0011                 | 3.2                         |
| 14           | 0.101 - 0.200             | 0.0012                 | 0.9                         |
| 2            | 0.201 - 0.50              | n.a.                   | n.a.                        |
| 9            | 0.501 - 1.00              | 0.0036                 | 0.6                         |
| 78           | OVERALL                   | 0.0014                 |                             |

## OTHER CHECKS:

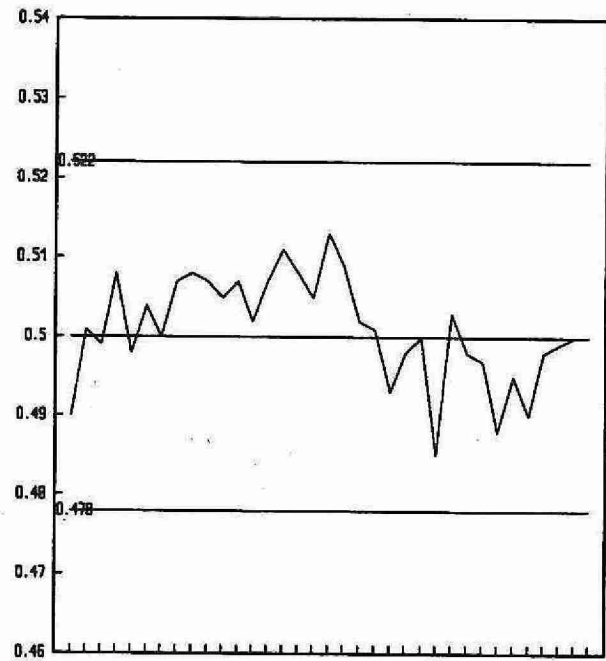
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 35 | 0.003 | 0.0095                 |

**SODIUM** (mg/L as Na)

QUALITY CONTROL DATA FROM 11/01/93 TO 12/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## SODIUM

### IDENTIFICATION:

|                      |                                      |                   |            |
|----------------------|--------------------------------------|-------------------|------------|
| Laboratory Unit      | Atomic Absorption                    | Method Introduced | 01/04/74   |
| Method Reference No. | E3171A                               | Units             | mg/L as Na |
| LIMS Product Code    | CAT3171,NAK3171                      | Supervisor        | J. McBride |
| Sample Type/Matrix   | Surface Waters, DWSP Drinking Waters |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.7 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 1.16 at the full scale level.

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.10 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 11 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA                   |
| Drift       | BL every 10 samples; 2 standards every 20 samples. |



# SODIUM

QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94

Laboratory Unit: Atomic Absorption

Full Scale: to 20.0 mg/L as Na

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 158 | 16.0                   | 16.1               | 0.1       | 0.1226                 |
| B:   | 158 | 4.00                   | 4.04               | 0.04      | 0.0621                 |
| C:   | 158 | 1.00                   | 1.01               | 0.01      | 0.0202                 |
| A+B: | 158 | 20.0                   | 20.1               | 0.1       | 0.1585                 |
| A-B: | 158 | 12.0                   | 12.1               | 0.1       | 0.1125                 |
| B+C: | 158 | 5.00                   | 5.04               | 0.04      | 0.0740                 |
| B-C: | 158 | 3.00                   | 3.03               | 0.03      | 0.0552                 |

s.d.(AB) S(between runs): 0.097

Sw(within run): 0.080

S/Sw: 1.2

s.d.(BC) S(between runs): 0.046

Sw(within run): 0.039

S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

|      |   |      |     |     |
|------|---|------|-----|-----|
| 19.4 | - | 20.6 | for | A+B |
| 11.6 | - | 12.4 | for | A-B |
| 4.70 | - | 5.30 | for | B+C |
| 2.80 | - | 3.20 | for | B-C |

## DUPLICATES:

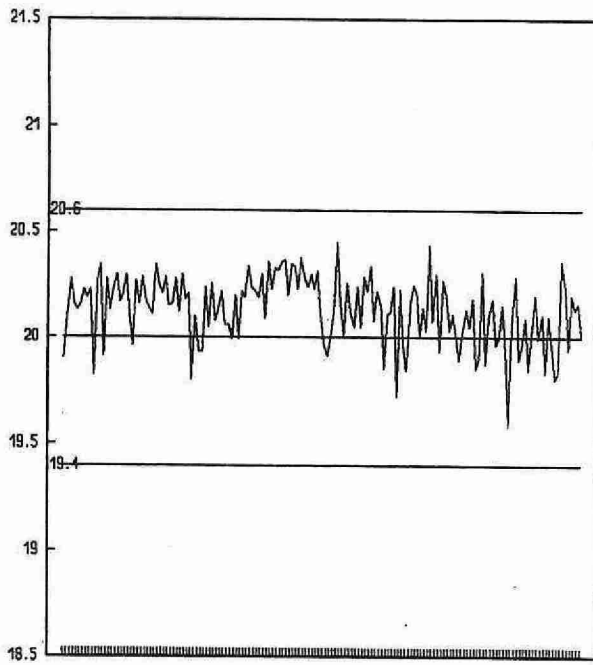
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 32           | 0.00 - 2.00               | 0.0178                 | 7.0                         |
| 43           | 2.01 - 4.00               | 0.0355                 | 1.5                         |
| 114          | 4.01 - 10.0               | 0.0683                 | 1.5                         |
| 104          | 10.0 - 20.0               | 0.1110                 | 0.8                         |
| 293          | Overall                   | 0.0738                 |                             |

## OTHER CHECKS:

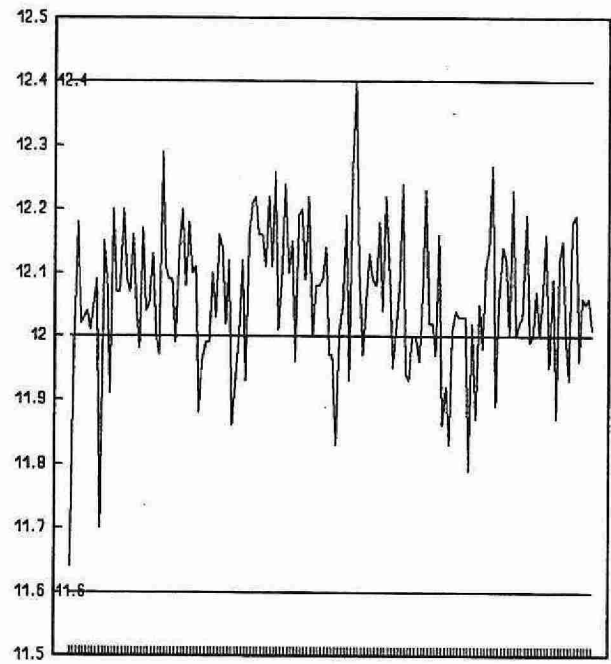
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 156 | -0.003 | 0.0124                 |

SODIUM (mg/L as Na)

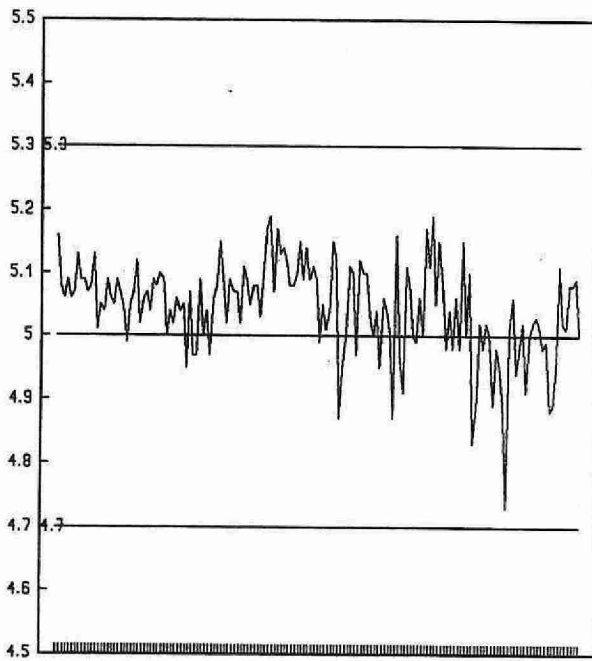
QUALITY CONTROL DATA FROM 06/01/94 TO 21/12/94



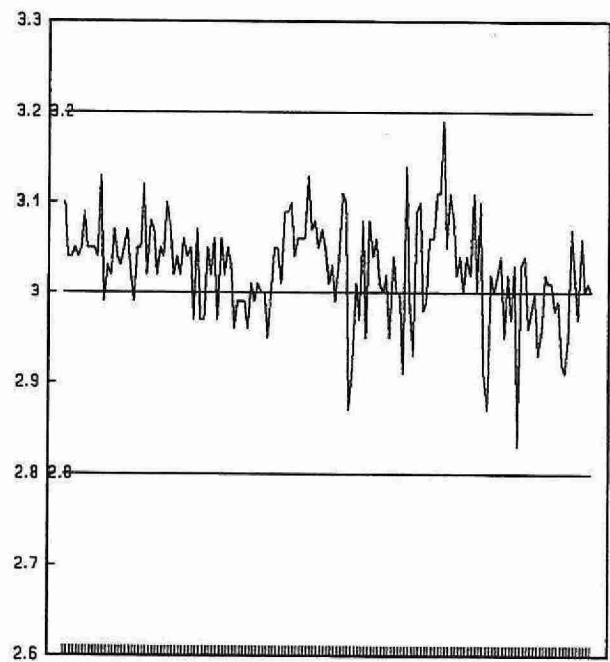
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## SODIUM

### IDENTIFICATION:

|                      |   |                   |            |
|----------------------|---|-------------------|------------|
| Laboratory Unit      | Atomic Absorption   | Method Introduced | 08/04/86   |
| Method Reference No. | E3217A  | Units             | mg/L as Na |
| LIMS Product Code    | CAT3217,CATS3217,NA3217   | Supervisor        | J. McBride |
| Sample Type/Matrix   | Domestic Waters, Leachates, Effluents, Sewage,Industrial Wastes |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 6 mL             |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 1.21 at the full scale level.

### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1.0 |
|--------------------------------|----------------------|----------------------|

### CALIBRATION:

BL plus 11 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA                   |
| Drift       | BL every 10 samples; 2 standards every 20 samples. |

# SODIUM

QUALITY CONTROL DATA FROM 04/01/94 TO 29/12/94

Laboratory Unit: Absorption

Full Scale: to 100.00 mg/L as Na

## CALIBRATION CONTROL:

|      | n   | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|-----|------------------------|--------------------|-----------|------------------------|
| A:   | 106 | 80.0                   | 80.02              | 0.02      | 1.1455                 |
| B:   | 106 | 20.0                   | 20.08              | 0.08      | 0.4307                 |
| C:   | 106 | 5.0                    | 5.02               | 0.03      | 0.2779                 |
| A+B: | 106 | 100.0                  | 100.11             | 0.11      | 1.4029                 |
| A-B: | 106 | 60.0                   | 59.94              | -0.06     | 1.0158                 |
| B+C: | 106 | 25.0                   | 25.11              | 0.11      | 0.6189                 |
| B-C: | 106 | 15.0                   | 15.06              | 0.06      | 0.3788                 |

s.d.(AB) S(between runs): 0.87

Sw(within run): 0.72

S/Sw: 1.2

s.d.(BC) S(between runs): 0.36

Sw(within run): 0.27

S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |        |     |     |
|-------|---|--------|-----|-----|
| 95.50 | - | 104.50 | for | A+B |
| 57.00 | - | 63.00  | for | A-B |
| 22.25 | - | 27.75  | for | B+C |
| 13.50 | - | 16.50  | for | B-C |

## DUPLICATES:

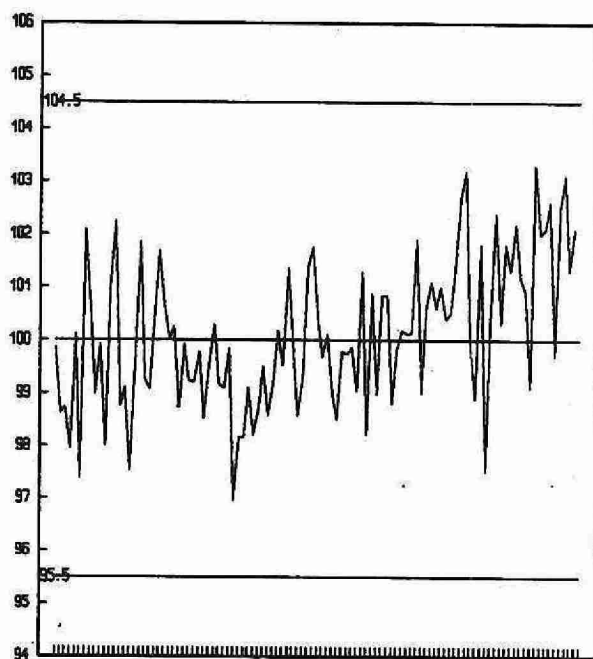
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 92           | 0.00 - 10.00              | 0.2164                 | 4.4                         |
| 55           | 10.01 - 25.00             | 0.2834                 | 2.0                         |
| 51           | 25.01 - 50.00             | 0.4918                 | 1.5                         |
| 47           | 50.01 - 100.00            | 1.2708                 | 1.6                         |
| 245          | Overall                   | 0.4365                 |                             |

## OTHER CHECKS:

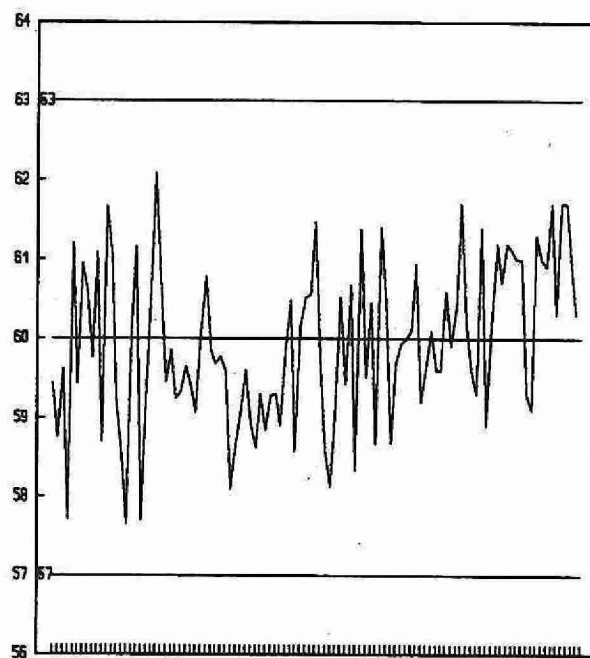
|                 | n   | Mean   | Standard Deviation (1) |
|-----------------|-----|--------|------------------------|
| Long Term Blank | 106 | -0.260 | 0.2768                 |

**SODIUM** (mg/L as Na)

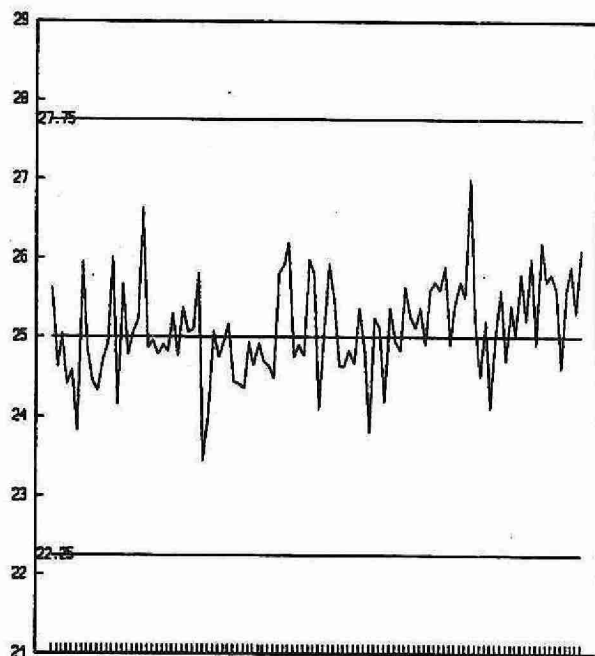
QUALITY CONTROL DATA FROM 04/01/94 TO 29/12/94



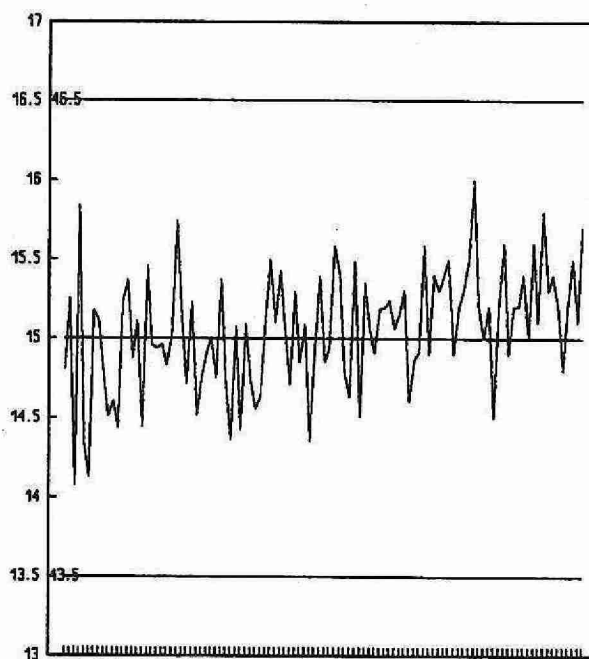
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT

## SODIUM

### IDENTIFICATION:

|                      |                |                   |            |
|----------------------|----------------|-------------------|------------|
| Laboratory Unit      | Dorset         | Method Introduced | 20/07/88   |
| LIS Test Name Code   | NAUR           | Units             | mg/L as Na |
| Work Station Code    | DOFLAME        | Unit Code         | 064811     |
| Method Code          | 001EA1         | Supervisor        | J. McBride |
| Method Reference No. | E3249A         |                   |            |
| Sample Type/Matrix   | Rivers, Lakes, |                   |            |

### SAMPLING:

|                   |         |
|-------------------|---------|
| Quantity Required | 5 mL    |
| Container         | Plastic |

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.005 | Current T value: 0.025 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

|             |  |
|-------------|--|
| Calibration | LTBL plus 3 standards, e.g., QCA       |
| Drift       | BL, reslope standard every 10 samples. |

### NOTES:

The control standards are corrected for the LTB from which they were made.

# SODIUM

QUALITY CONTROL DATA FROM 13/01/94 TO 22/12/94

Laboratory Unit: Dorset

Full Scale: to 4.0 mg/L as Na

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 75 | 3.2                    | 3.211              | 0.011     | 0.0245                 |
| B:   | 75 | 0.8                    | 0.801              | 0.001     | 0.0091                 |
| C:   | 75 | 0.2                    | 0.204              | 0.004     | 0.0045                 |
| A+B: | 75 | 4.0                    | 4.008              | 0.008     | 0.0264                 |
| A-B: | 75 | 2.4                    | 2.410              | 0.010     | 0.0252                 |
| B+C: | 75 | 1.0                    | 1.001              | 0.001     | 0.0106                 |
| B-C: | 75 | 0.6                    | 0.597              | -0.003    | 0.0101                 |

s.d.(AB) S(between runs): 0.018

Sw(within run): 0.018

S/Sw: 1.04

s.d.(BC) S(between runs): 0.007

Sw(within run): 0.007

S/Sw: 1.01

The calibration is accepted if the calibration control values obtained lie within the ranges:

|       |   |       |     |     |
|-------|---|-------|-----|-----|
| 3.91  | - | 4.09  | for | A+B |
| 2.33  | - | 2.47  | for | A-B |
| 0.958 | - | 1.04  | for | B+C |
| 0.568 | - | 0.632 | for | B-C |

## DUPLICATES:

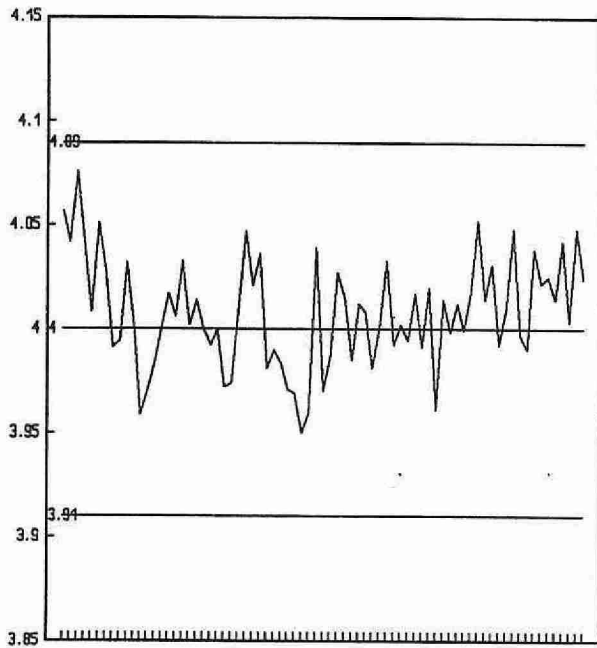
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 72           | 0.00 - 0.40               | 0.0058                 | 6.4                         |
| 77           | 0.41 - 0.80               | 0.0159                 | 3.4                         |
| 46           | 0.81 - 2.00               | 0.0379                 | 4.2                         |
| 19           | 2.01 - 4.00               | 0.0726                 | 3.6                         |
| 214          | Overall                   | 0.0179                 |                             |

## OTHER CHECKS:

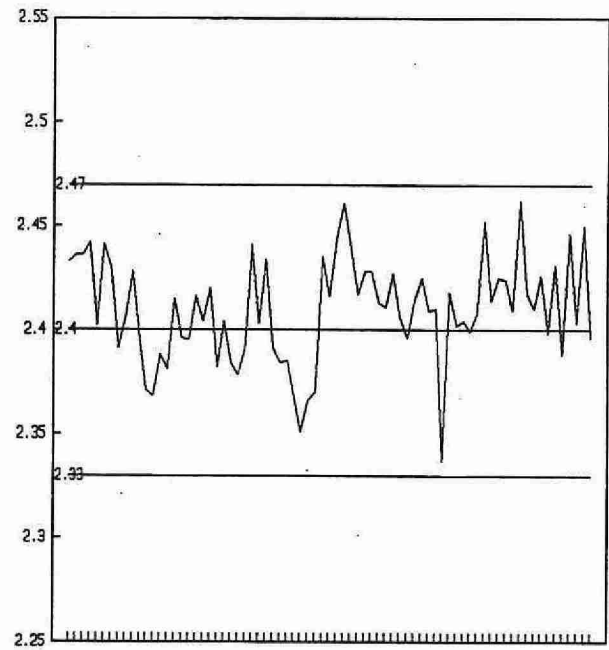
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 75 | 0.0020 | 0.0042                 |

SODIUM (mg/L as Na)

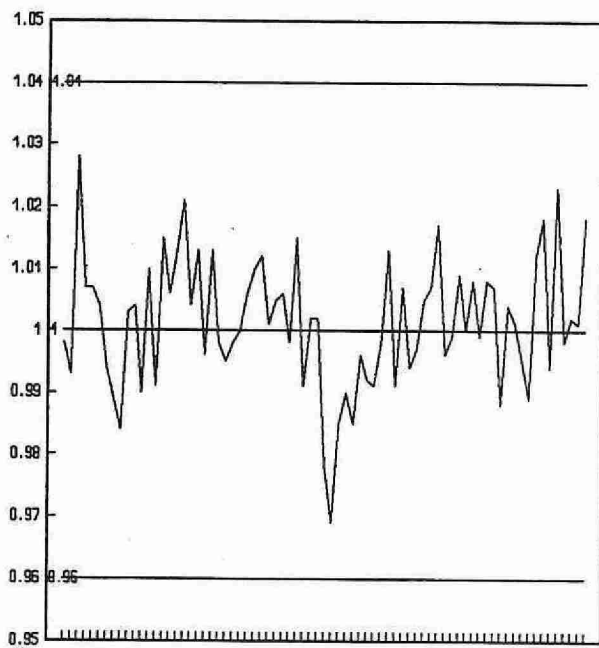
QUALITY CONTROL DATA FROM 13/01/94 TO 22/12/94



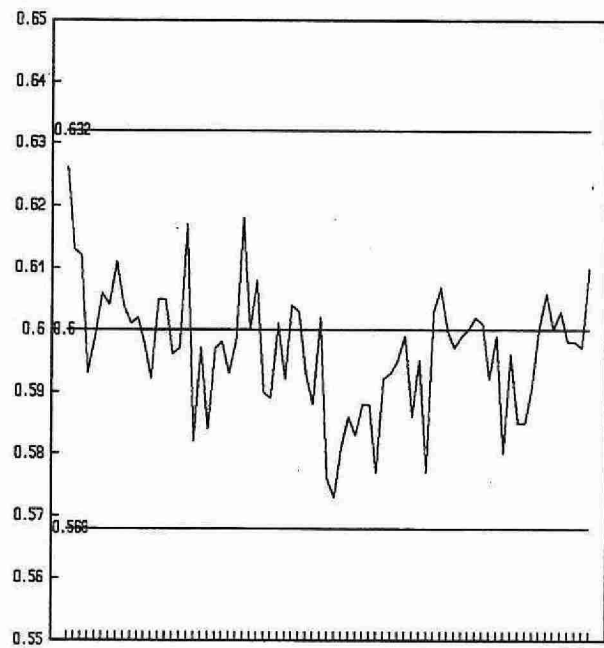
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD B+C



QUALITY CONTROL STANDARD B-C

CONTROL LIMIT



## SOLIDS, DISSOLVED

### IDENTIFICATION:

|                      |                          |                   |            |
|----------------------|--------------------------|-------------------|------------|
| Laboratory Unit      | Solids                   | Method Introduced | Before '61 |
| Method Reference No. | E3188B                   | Units             | mg/L       |
| LIMS Product Code    | TSD3188,DS3188,DIGN3188  | Supervisor        | J. McBride |
| Sample Type/Matrix   | Sewage, Industrial Waste |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 125 mL           |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Sample is filtered under moderate suction through a Whatman 934AH grade glass fibre filter. Generally 100 mL of filtrate (alternate 50 mL) is pipetted into a preweighed Teflon dish, dried at 103  $\pm$  2  $^{\circ}$ C, and stored in a desiccator for at least 24 hours. The dissolved solids content is calculated by subtracting the original dish mass from the dried residue + dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

### INSTRUMENTATION:

Balance (5 decimal places), drying oven, suction filtration apparatus, dishes (Teflon)  
Microcomputer system with appropriate software

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |
|--------------------------------|--------------------|---------------------|

### CALIBRATION:

Balance zero  
Balance internal calibration is performed daily.

### CONTROLS:

|              |   |
|--------------|---|
| Calibration  | 2 S class weights, e.g. QCA (results in grams)                          |
| Drift        | Balance is reset to zero after every 10 weighings by the microcomputer. |
| Recovery     | 2 standards, e.g. R1  |
| Method Blank | 100 mL distilled water.   |

### NOTES:

In June 1994, Solids method E3188B was written to include methods for all Solids fractions (Total, Suspended, Dissolved and Ignited) on sewage and industrial waste samples. The former methods which covered these techniques for all matrices, were discontinued ie., E3188A, E3190A, E3192A, and E3194A.

# SOLIDS, DISSOLVED

QUALITY CONTROL DATA FROM 01/01/94 TO 31/12/94

Laboratory Unit: Solids

## CALIBRATION CONTROL:

|      | n  | Expected Mass (g) | Av. Mass Measured (g) | Av. Bias (g) | Standard Deviation (1) |
|------|----|-------------------|-----------------------|--------------|------------------------|
| A:   | 89 | 50.00             | 50.00001              | 0.00001      | 0.000073               |
| B:   | 89 | 30.00             | 30.0001               | 0.0001       | 0.000063               |
| A+B: | 89 | 80.00             | 80.00011              | 0.00011      | 0.000123               |
| A-B: | 89 | 20.00             | 19.99991              | -0.00009     | 0.000058               |

s.d.(AB) S(between runs): 0.000068 Sw(within run): 0.000041 S/Sw: 1.7

The calibration is accepted if the calibration control values obtained lie within the ranges expressed in grams:

79.9996 - 80.0004 for A+B  
19.9997 - 20.0003 for A-B

## RECOVERIES:

| Number of Data | Expected Conc (mg/L) | Av. Conc Measured (mg/L) | Standard Deviation (1) |
|----------------|----------------------|--------------------------|------------------------|
| 86             | 2000.0               | 1996.82                  | 6.0959                 |
| 88             | 500.0                | 498.92                   | 3.9729                 |

## DUPLICATES:

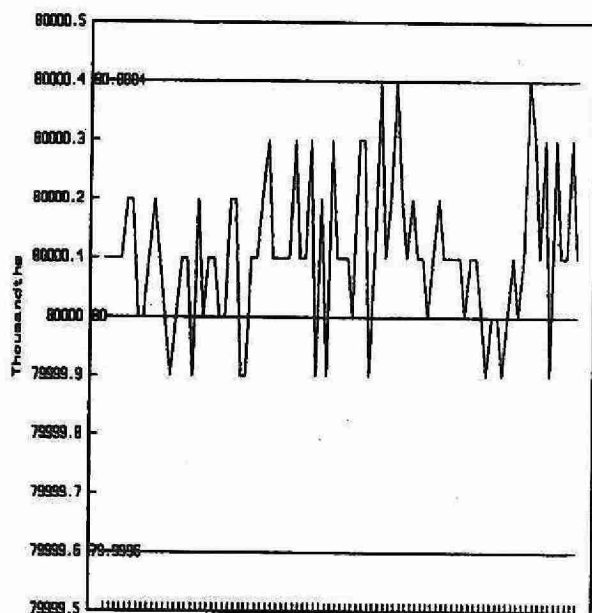
| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|----------------------------------|------------------------|-----------------------------|
| 82           | 0 - 500                          | 3.7070                 | 1.53                        |
| 60           | 501 - 1000                       | 6.2719                 | 1.54                        |
| 48           | 1001 - 5000                      | 15.8645                | 2.36                        |
| 4            | 5001 - 10000                     | 35.1864                | 0.53                        |
| 194          | Overall                          | 6.6490                 |                             |

## OTHER CHECKS:

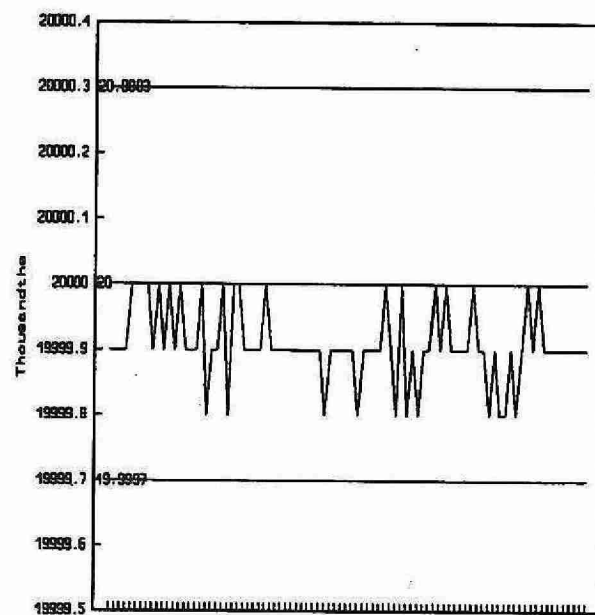
|       | n  | Data Mean (mg/L) | Standard Deviation (1) |
|-------|----|------------------|------------------------|
| Blank | 89 | 0.818            | 3.2285                 |

# SOLIDS, DISSOLVED (mg/L)

QUALITY CONTROL DATA FROM 01/01/94 TO 31/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## SOLIDS, DISSOLVED

### IDENTIFICATION:

|                      |  |                   |            |
|----------------------|--|-------------------|------------|
| Laboratory Unit      | Solids River                               | Method Introduced | Before '61 |
| Method Reference No. | E3365A                                     | Units             | mg/L       |
| LIMS Product Code    | TSD3365,DS3365                             | Supervisor        | J. McBride |
| Sample Type/Matrix   | Domestic Waters, Surface Waters, Leachates |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 125 mL           |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Sample is filtered under moderate suction through a Whatman 934AH grade glass fibre filter. Generally 100 mL of filtrate (alternate 50 mL) is pipetted into a preweighed Teflon dish, dried at 103-105°C, and stored in a desiccator for at least 24 hours. The dissolved solids content is calculated by subtracting the original dish mass from the dried dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system. Dissolved solids may be calculated, if the conductivity is less than 800 S by the formula:

$$\text{Dissolved Solids} = \text{Conductivity} * 0.65$$

### INSTRUMENTATION:

Balance (5 decimal places), drying oven, suction filtration apparatus, dishes (Teflon)  
Microcomputer system with appropriate software

### REPORTING:

|                                |                    |                     |
|--------------------------------|--------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |
|--------------------------------|--------------------|---------------------|

### CALIBRATION:

Balance zero  
Balance internal calibration is performed daily.

### CONTROLS:

|              |   |
|--------------|---|
| Calibration  | 2 S class weights, e.g. QCA (results in grams)                          |
| Drift        | Balance is reset to zero after every 10 weighings by the microcomputer. |
| Recovery     | 2 standards, e.g. R1  |
| Method Blank | 100 mL distilled water.   |

### NOTES:

In June 1994, Solids method E3365A was written to include methods for all Solids fractions (Total, Suspended, and Dissolved) on surface and drinking waters. The former methods which covered these techniques for all matrices, were discontinued ie., E3188A, E3190A, and E3192A.

# SOLIDS, DISSOLVED

QUALITY CONTROL DATA FROM 10/01/94 TO 25/11/94

Laboratory Unit: River Solids

## CALIBRATION CONTROL:

|      | n  | Expected Mass (g) | Av. Mass Measured (g) | Av. Bias (g) | Standard Deviation (1) |
|------|----|-------------------|-----------------------|--------------|------------------------|
| A:   | 90 | 50.00             | 50.00049              | -0.00001     | 0.000088               |
| B:   | 90 | 30.00             | 30.00030              | 0.00030      | 0.000080               |
| A+B: | 90 | 80.00             | 80.00080              | 0.00080      | 0.000154               |
| A-B: | 90 | 20.00             | 20.00019              | 0.00019      | 0.000066               |

s.d.(AB) S(between runs): 0.000084 Sw(within run): 0.000047 S/Sw: 1.8

The calibration is accepted if the calibration control values obtained lie within the ranges expressed in grams:

80.00045 - 80.00114 for A+B  
19.99994 - 20.00046 for A-B

## RECOVERIES:

| Number of Data | Expected Conc (mg/L) | Av. Conc Measured (mg/L) | Standard Deviation (1) |
|----------------|----------------------|--------------------------|------------------------|
| 65             | 2000.0               | 2003.0                   | 15.8                   |
| 66             | 500.0                | 496.8                    | 9.3                    |

## DUPLICATES:

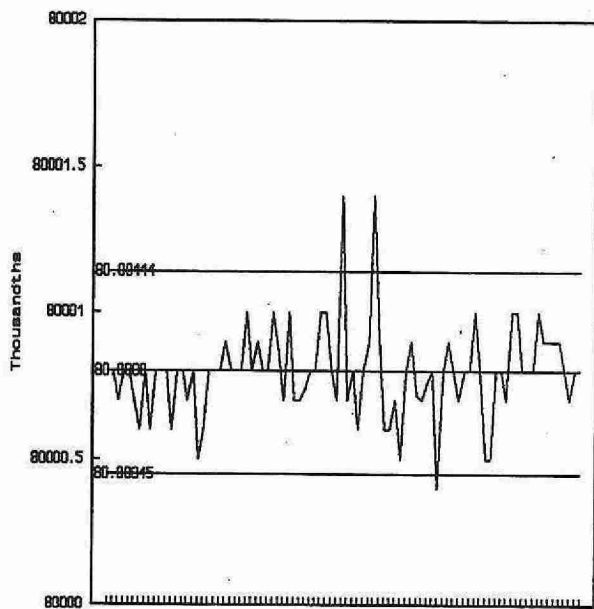
| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|----------------------------------|------------------------|-----------------------------|
| 16           | 0.0 - 500.0                      | 8.3432                 | 3.5                         |
| 136          | 500.1 - 1000.0                   | 8.7267                 | 1.6                         |
| 18           | 1000.1 - 5000.0                  | 12.1793                | 0.9                         |
| 170          | OVERALL                          | 9.1341                 |                             |

## OTHER CHECKS:

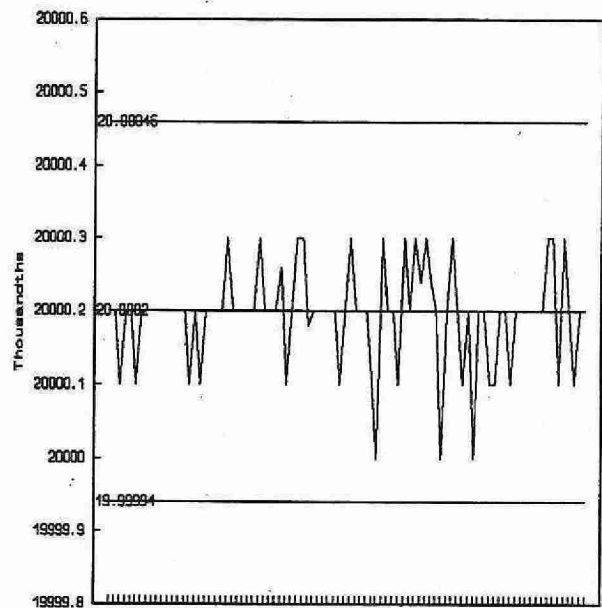
|       | n  | Data Mean (mg/L) | Standard Deviation (1) |
|-------|----|------------------|------------------------|
| Blank | 68 | -1.882           | 14.8                   |

# SOLIDS, DISSOLVED (mg/L)

QUALITY CONTROL DATA FROM 10/01/94 TO 25/11/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## SOLIDS, SUSPENDED

### IDENTIFICATION:

|                      |                          |                   |            |
|----------------------|--------------------------|-------------------|------------|
| Laboratory Unit      | Solids                   | Method Introduced | Before '81 |
| Method Reference No. | E3188B                   | Units             | mg/L       |
| LIMS Product Code    | TSD3188,SS3188,SIGN3188  | Supervisor        | J. McBride |
| Sample Type/Matrix   | Sewage, Industrial Waste |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 5-500 mL         |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

An appropriate shaken sample volume (5 to 500 mL) is pipetted or quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a preweighed Whatman 934AH glass fibre filter. The graduated cylinder and then the filter are washed with a total of 50 mL distilled water. The filter is dried at 103-105°C, and suspended solids content is calculated by subtracting the original filter mass from the dried filter mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

### INSTRUMENTATION:

Balance (5-decimal places), drying oven, suction filtration apparatus  
Microcomputer system with appropriate software

### REPORTING:

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |
|--------------------------------|----------------------|----------------------|

### CONTROLS:

|              |   |
|--------------|---|
| Calibration  | 2 S class weights, e.g. QCA (results in grams)                          |
| Drift        | Balance is reset to zero after every 10 weighings by the microcomputer. |
| Recovery     | 2 standards, e.g. R1  |
| Method Blank | Filter washed with 500 mL distilled water                               |

### NOTES:

A standard correction factor was applied to all filters to account for weight loss during filtering (-0.0003g).

In June 1994, Solids method E3188B was written to include methods for all Solids fractions (Total, Suspended, Dissolved and Ignited) on sewage and industrial waste samples. The former methods which covered these techniques for all matrices, were discontinued ie., E3188A, E3190A, E3192A, and E3194A.



# SOLIDS, SUSPENDED

QUALITY CONTROL DATA FROM 01/01/94 TO 31/12/94

Laboratory Unit: Solids

CALIBRATION CONTROL: (QC data from SS3188 and SIGN3188 )

|      | n   | Expected Mass (g) | Av. Mass Measured (g) | Av. Bias (g) | Standard Deviation (1) |
|------|-----|-------------------|-----------------------|--------------|------------------------|
| A:   | 190 | 0.50              | 0.49999               | -0.00001     | 0.000010               |
| B:   | 190 | 0.05              | 0.04999               | -0.00001     | 0.000013               |
| A+B: | 190 | 0.55              | 0.54999               | -0.00001     | 0.000019               |
| A-B: | 190 | 0.45              | 0.44999               | -0.00001     | 0.000014               |

s.d.(AB) S(between runs): 0.000012 Sw(within run): 0.0000097 S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges expressed in

grams:

0.54992 - 0.55008 for A+B

0.44994 - 0.45006 for A-B

RECOVERIES:

| Number of Data | Expected Conc (mg/L) | Av. Conc Measured (mg/L) | Standard Deviation (1) |
|----------------|----------------------|--------------------------|------------------------|
| 273            | 200.0                | 194.45                   | 1.9279                 |
| 272            | 50.0                 | 49.004                   | 0.8533                 |

DUPLICATES:

| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|----------------------------------|------------------------|-----------------------------|
| 53           | 0 - 10                           | 1.0920                 | 18.3                        |
| 84           | 10 - 25                          | 1.1943                 | 7.7                         |
| 228          | 25 - 100                         | 2.7868                 | 5.3                         |
| 180          | 100 - 500                        | 8.5849                 | 8.7                         |
| 13           | 500 - 1000                       | 13.7828                | 2.6                         |
| 125          | 1000 - 10000                     | 45.6063                | 3.8                         |
| 3            | 10000 - 30000                    | 622.8671               | 2.7                         |
| 686          | Overall                          | 6.8136                 |                             |

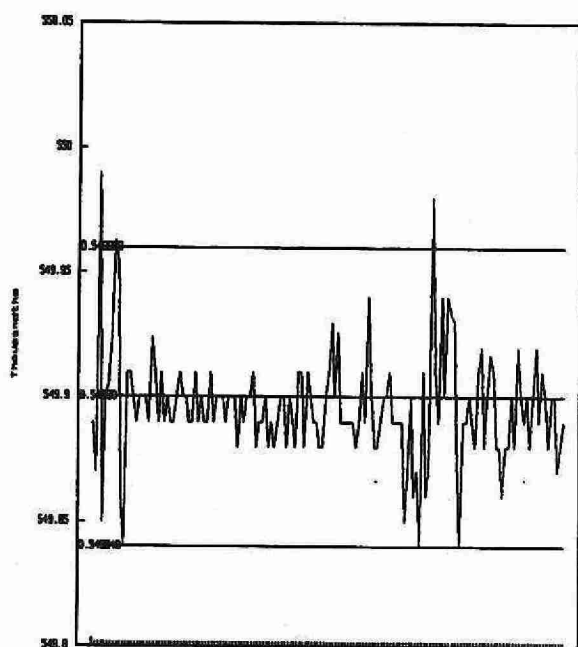
OTHER CHECKS:

|       | n   | Data Mean (mg/L) | Standard Deviation (1) |
|-------|-----|------------------|------------------------|
| Blank | 274 | 0.1005           | 0.1863                 |

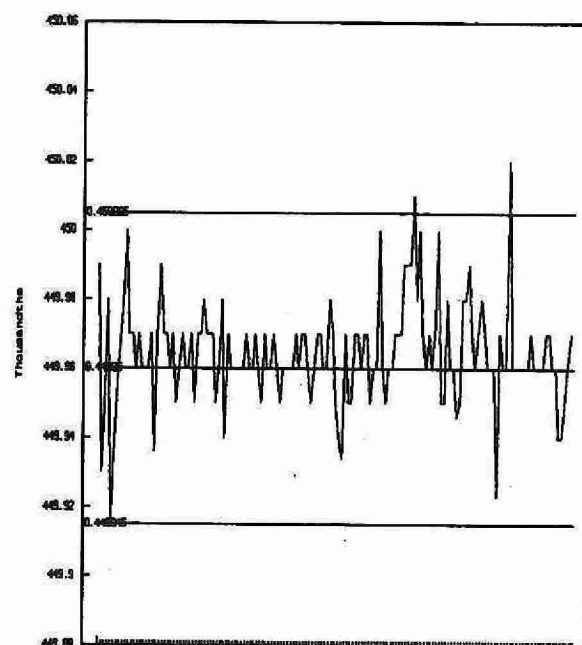


# SOLIDS, SUSPENDED (mg/L)

QUALITY CONTROL DATA FROM 10/01/94 TO 31/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## SOLIDS, SUSPENDED

### IDENTIFICATION:

|                      |   |                   |            |
|----------------------|---|-------------------|------------|
| Laboratory Unit      | Solids River  | Method Introduced | Before '81 |
| Method Reference No. | E3365A  | Units             | mg/L       |
| LIMS Product Code    | TSD3365,SS3365  | Supervisor        | J. McBride |
| Sample Type/Matrix   | Drinking Waters, Leachates, Effluents, Surface Waters |                   |            |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 5-500 mL         |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

An appropriate shaken sample volume (5 to 500 mL) is pipetted or quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a preweighed Whatman 934AH glass fibre filter. The graduated cylinder and then the filter are washed with a total of 30 mL distilled water. The filter is dried at 103-105°C, and suspended solids content is calculated by subtracting the original filter mass from the dried filter mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

### INSTRUMENTATION:

Balance (5-decimal places), drying oven, suction filtration apparatus  
Microcomputer system with appropriate software

### REPORTING:

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |
|--------------------------------|----------------------|----------------------|

### CONTROLS:

|              |   |
|--------------|---|
| Calibration  | 2 S class weights, e.g. QCA (results in grams)                          |
| Drift        | Balance is reset to zero after every 10 weighings by the microcomputer. |
| Recovery     | 2 standards, e.g. R1  |
| Method Blank | Filter washed with 500 mL distilled water                               |

### NOTES:

In June 1994, Solids method E3365A was written to include methods for all Solids fractions (Total, Suspended, and Dissolved) on surface and drinking waters. The former methods which covered these techniques for all matrices, were discontinued ie., E3188A, E3190A, and E3192A.

# SOLIDS, SUSPENDED

QUALITY CONTROL DATA FROM 10/01/94 TO 31/12/94

Laboratory Unit: River Solids

## CALIBRATION CONTROL:

|      | n   | Expected Mass (g) | Av. Mass Measured (g) | Av. Bias (g) | Standard Deviation (1) |
|------|-----|-------------------|-----------------------|--------------|------------------------|
| A:   | 155 | 0.50              | 0.499930              | -0.000070    | 0.000011               |
| B:   | 155 | 0.05              | 0.049966              | -0.000034    | 0.000015               |
| A+B: | 155 | 0.55              | 0.549896              | -0.000104    | 0.000022               |
| A-B: | 155 | 0.45              | 0.449964              | -0.000036    | 0.000015               |

s.d.(AB) S(between runs): 0.000013 Sw(within run): 0.00001 S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges expressed in

grams:  
 0.549840 - 0.549960 for A+B  
 0.449915 - 0.450005 for A-B

## RECOVERIES:

| Number of Data | Expected Conc (mg/L) | Av. Conc Measured (mg/L) | Standard Deviation (1) |
|----------------|----------------------|--------------------------|------------------------|
| 146            | 200.0                | 193.48                   | 3.6533                 |
| 145            | 50.0                 | 48.97                    | 2.2044                 |

## DUPLICATES:

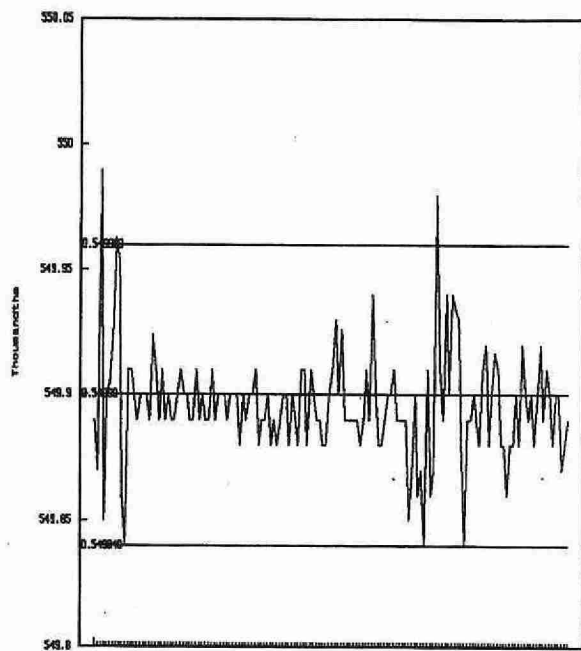
| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|----------------------------------|------------------------|-----------------------------|
| 148          | 0.0 - 50.0                       | 1.7040                 | 13.3                        |
| 120          | 50.1 - 200.0                     | 3.8169                 | 5.2                         |
| 45           | 200.1 - 1000.0                   | 13.7606                | 9.5                         |
| 10           | 1000.1 - 5000.0                  | 79.4119                | 6.4                         |
| 323          | OVERALL                          | 4.0016                 |                             |

## OTHER CHECKS:

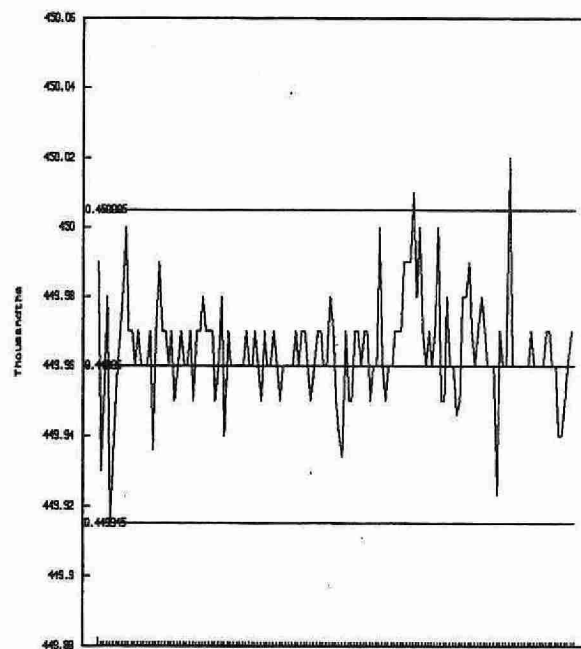
|       | n   | Data Mean (mg/L) | Standard Deviation (1) |
|-------|-----|------------------|------------------------|
| Blank | 146 | 0.1063           | 0.7308                 |

# SOLIDS, SUSPENDED (mg/L)

QUALITY CONTROL DATA FROM 10/01/94 TO 31/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

**SOLIDS, SUSPENDED IGNITED**  
(Particulate Ash and Particulate Loss On Ignition)

**IDENTIFICATION:**

|                      |                          |                   |            |
|----------------------|--------------------------|-------------------|------------|
| Laboratory Unit      | Solids                   | Method Introduced | Before '61 |
| Method Reference No. | E3188B                   | Units             | mg/L       |
| LIMS Product Code    | SIGN3188                 | Supervisor        | J. McBride |
| Sample Type/Matrix   | Sewage, Industrial Waste |                   |            |

**SAMPLING:**

|                   |                  |
|-------------------|------------------|
| Quantity Required | 5-500 mL         |
| Container         | Glass or plastic |

**ANALYTICAL PROCEDURE:**

The procedure for particulate solids (SS3188) is followed and the dried residue is ignited at 600 50 °C for one hour in a muffle furnace. As soon as practical, the dish is transferred to a desiccator to cool. The particulate ash (fixed solids) is the difference between the final ignited mass plus filter and the original tare weight of the filter, divided by the original sample volume (mL) used for SS3188. The particulate loss on ignition (estimate of volatile suspended solids) is the difference between the final ignited mass plus filter and the residue (suspended solids) plus filter, divided by the original sample volume (mL). Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

**INSTRUMENTATION:**

Balance (5 decimal places), muffle furnace, filters, Petri dishes  
Microcomputer system with appropriate software

**REPORTING:**

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |
|--------------------------------|----------------------|----------------------|

**CONTROLS:**

|             |   |
|-------------|---|
| Calibration | 2 S class weights, e.g. QCA (results in grams)                          |
| Drift       | Balance is reset to zero after every 10 weighings by the microcomputer. |

**NOTES:**

In June 1994, Solids method E3188B was written to include methods for all Solids fractions (Total, Suspended, Dissolved and Ignited) on sewage and industrial waste samples. The former methods which covered these techniques for all matrices, were discontinued ie., E3188A, E3190A, E3192A, and E3194A.

**SOLIDS, SUSPENDED IGNITED**  
(Particulate Ash and Particulate Loss On Ignition)

QUALITY CONTROL DATA FROM 01/01/94 TO 31/12/94

Laboratory Unit: Solids

**CALIBRATION CONTROL:**

|      | n  | Expected Mass (g) | Av. Mass Measured (g) | Av. Bias (g) | Standard Deviation (1) |
|------|----|-------------------|-----------------------|--------------|------------------------|
| A:   | 88 | 0.50              | 0.499998              | -0.000002    | 0.000009               |
| B:   | 88 | 0.05              | 0.049997              | -0.000003    | 0.000009               |
| A+B: | 88 | 0.55              | 0.549995              | -0.000005    | 0.000016               |
| A-B: | 88 | 0.45              | 0.450000              | -0.000000    | 0.000009               |

s.d.(AB)      S(between runs): 0.000009      Sw(within run): 0.000007      S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges expressed in grams:

0.54992 - 0.55008 for A+B  
0.44994 - 0.45006 for A-B

**SOLIDS, SUSPENDED IGNITED ( PARTICULATE ASH )**

**DUPLICATES:**

| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|----------------------------------|------------------------|-----------------------------|
| 48           | 0 - 10.0                         | 0.4186                 | 11.0                        |
| 49           | 10.1 - 50.0                      | 1.2268                 | 7.1                         |
| 23           | 50.1 - 500.0                     | 8.6713                 | 8.4                         |
| 38           | 500.1 - 1000.0                   | 12.4644                | 1.8                         |
| 61           | 1000.1 - 5000.0                  | 19.6237                | 4.4                         |
| 219          | OVERALL                          | 8.0649                 |                             |

**OTHER CHECKS:**

|       | n  | Data Mean (mg/L) | Standard Deviation (1) |
|-------|----|------------------|------------------------|
| Blank | 88 | -0.2814          | 0.2335                 |

**SOLIDS, SUSPENDED IGNITED**  
**(Particulate Ash and Particulate Loss On Ignition)**

**SOLIDS, SUSPENDED IGNITED ( PARTICULATE LOSS ON IGNITION )**

**DUPLICATES:**

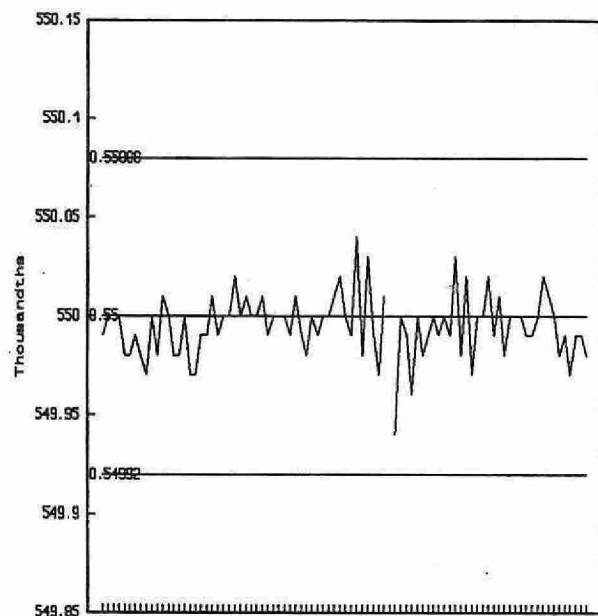
| n<br>Data Pairs | Sample<br>Concentration Span (mg/L) | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|-------------------------------------|---------------------------|--------------------------------|
| 33              | 0 - 10.0                            | 0.4247                    | 12.5                           |
| 47              | 10.1 - 50.0                         | 1.2198                    | 6.9                            |
| 28              | 50.1 - 500.0                        | 3.1185                    | 3.4                            |
| 10              | 500.1 - 1000.0                      | 14.2581                   | 1.9                            |
| 101             | 1000.1 - 15000.0                    | 30.9118                   | 3.6                            |
| 219             | OVERALL                             | 13.2251                   |                                |

**OTHER CHECKS:**

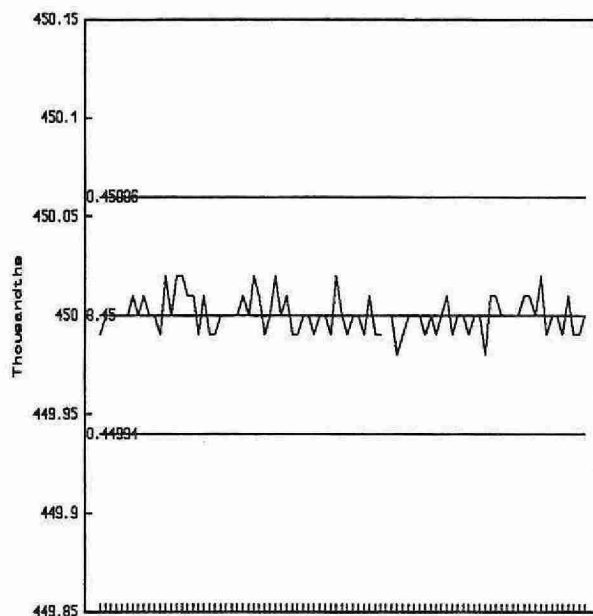
|       | n  | Data<br>Mean (mg/L) | Standard<br>Deviation (1) |
|-------|----|---------------------|---------------------------|
| Blank | 88 | 0.4127              | 0.1361                    |

**SOLIDS, SUSPENDED IGNITED (mg/L)**  
**(Particulate Ash and Particulate Loss on Ignition)**

QUALITY CONTROL DATA FROM 01/01/94 TO 31/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT



## SOLIDS, TOTAL

### IDENTIFICATION:

|                      |                          |                   |               |
|----------------------|--------------------------|-------------------|---------------|
| Laboratory Unit      | Solids                   | Method Introduced | Before '81    |
| Method Reference No. | E3188B                   | Units             | mg/L or mg/Kg |
| LIMS Product Code    | TSD3188,TS3188,TIGN3188  | Supervisor        | J. McBride    |
| Sample Type/Matrix   | Sewage, Industrial Waste |                   |               |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 125 mL           |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Generally, 100 mL aliquot of sample (alternate 50 mL) is pipetted into a preweighed Teflon dish, dried at 103-105°C, and stored in a desiccator for at least 24 hours. The total residue or solids content is calculated by subtracting the original dish mass from the dried dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

### INSTRUMENTATION:

Balance (5 decimal places), drying oven, dishes (Teflon)  
Microcomputer system with appropriate software

### REPORTING:

|                                |                      |                     |
|--------------------------------|----------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2.0 | Current T value: 10 |
|--------------------------------|----------------------|---------------------|

### CALIBRATION:

Balance zero  
Balance internal calibration performed daily.

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | 2 S class weights, e.g. QCA (results in grams)                          |
| Drift       | Balance is reset to zero after every 10 weighings by the microcomputer. |
| Recovery    | 2 standards, e.g. R1  |

### NOTES:

In June 1994, Solids method E3188B was written to include methods for all Solids fractions (Total, Suspended, Dissolved and Ignited) on sewage and industrial waste samples. The former methods which covered these techniques for all matrices, were discontinued ie., E3188A, E3190A, E3192A, and E3194A.

# SOLIDS, TOTAL

QUALITY CONTROL DATA FROM 01/01/94 TO 31/12/94

Laboratory Unit: Solids

CALIBRATION CONTROL: (QC data from TS3188 + TIGN3188)

|      | n  | Expected Mass (g) | Av. Mass Measured (g) | Av. Bias (g) | Standard Deviation (1) |
|------|----|-------------------|-----------------------|--------------|------------------------|
| A:   | 54 | 50.00             | 50.00001              | 0.00001      | 0.000072               |
| B:   | 54 | 30.00             | 30.00011              | 0.00011      | 0.000063               |
| A+B: | 54 | 80.00             | 80.00011              | 0.00011      | 0.000123               |
| A-B: | 54 | 20.00             | 19.99991              | -0.00009     | 0.000056               |

s.d.(AB)    S(between runs): 0.000068    Sw(within run): 0.000039    S/Sw: 1.7

The calibration is accepted if the calibration control values obtained lie within the ranges expressed in grams:

79.9996 - 80.0004 for A+B  
19.9997 - 20.0003 for A-B

## RECOVERIES:

| Number of Data | Expected Conc (mg/L) | Av. Conc Measured (mg/L) | Standard Deviation (1) |
|----------------|----------------------|--------------------------|------------------------|
| 49             | 20000.0              | 20039.2                  | 139.4                  |
| 52             | 2000.0               | 1998.1                   | 8.8132                 |

## DUPLICATES:

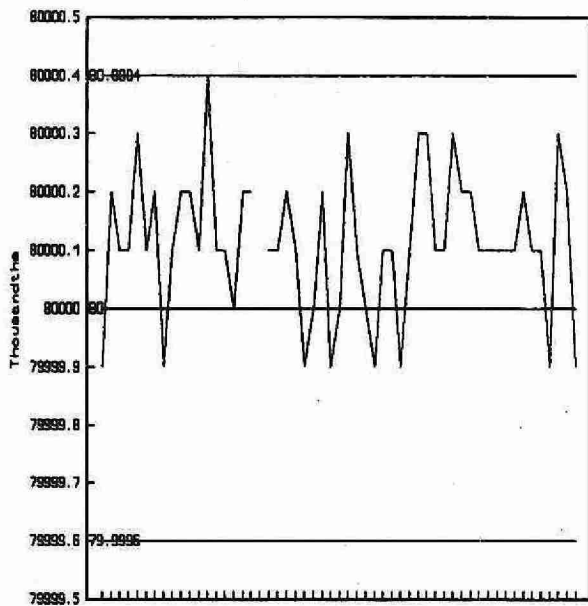
| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|----------------------------------|------------------------|-----------------------------|
| 9            | 0 - 1000                         | 5.7379                 | 1.7                         |
| 32           | 1001 - 5000                      | 32.9156                | 1.1                         |
| 32           | 5001 - 25000                     | 113.0477               | 1.4                         |
| 27           | 25001 - 60000                    | 289.0379               | 0.8                         |
| 100          | Overall                          | 94.8942                |                             |

## OTHER CHECKS:

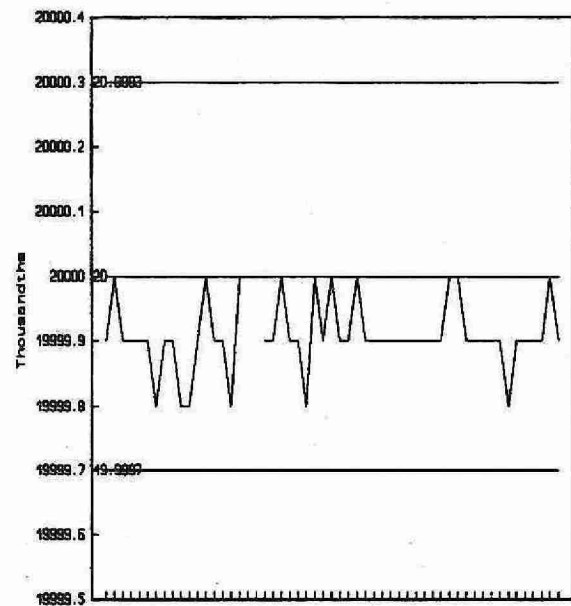
|       | n  | Data Mean (mg/L) | Standard Deviation (1) |
|-------|----|------------------|------------------------|
| Blank | 55 | 1.551            | 3.6764                 |

# SOLIDS, TOTAL (mg/L or mg/Kg)

QUALITY CONTROL DATA FROM 01/01/94 TO 31/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## SOLIDS, TOTAL

### IDENTIFICATION:

|                      |                             |                   |               |
|----------------------|-----------------------------|-------------------|---------------|
| Laboratory Unit      | Solids River                | Method Introduced | Before '81    |
| Method Reference No. | E3365A                      | Units             | mg/L or mg/Kg |
| LIMS Product Code    | TSD3365,TS3365              | Supervisor        | J. McBride    |
| Sample Type/Matrix   | Drinking Waters, Leachates, |                   |               |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 125 mL           |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Generally, 100 mL aliquot of sample (alternate 50 mL) is pipetted into a preweighed dish, dried at 103-105°C, and stored in a desiccator for at least 24 hours. The total residue or solids content is calculated by subtracting the original dish mass from the dried dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

### INSTRUMENTATION:

Balance (5 decimal places), drying oven, dishes (Teflon)  
Microcomputer system with appropriate software

### REPORTING:

|                                |                      |                     |
|--------------------------------|----------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2.0 | Current T value: 10 |
|--------------------------------|----------------------|---------------------|

### CALIBRATION:

Balance zero  
Balance internal calibration performed daily.

### CONTROLS:

|             |   |
|-------------|---|
| Calibration | 2 S class weights, e.g. QCA (results in grams)                          |
| Drift       | Balance is reset to zero after every 10 weighings by the microcomputer. |
| Recovery    | 2 standards, e.g. R1  |

### NOTES:

In June 1994, Solids method E3365A was written to include methods for all Solids fractions (Total, Suspended, and Dissolved) on surface and drinking waters. The former methods which covered these techniques for all matrices, were discontinued ie., E3188A, E3190A, and E3192A.

# SOLIDS, TOTAL

QUALITY CONTROL DATA FROM 14/01/94 TO 30/12/94

Laboratory Unit: River Solids

## CALIBRATION CONTROL:

|      | n  | Expected Mass (g) | Av. Mass Measured (g) | Av. Bias (g) | Standard Deviation (1) |
|------|----|-------------------|-----------------------|--------------|------------------------|
| A:   | 33 | 50.00             | 50.00046              | 0.00046      | 0.000094               |
| B:   | 33 | 30.00             | 30.00030              | 0.00030      | 0.000047               |
| A+B: | 33 | 80.00             | 80.00075              | 0.00075      | 0.000120               |
| A-B: | 33 | 20.00             | 20.00016              | 0.00016      | 0.000086               |

s.d.(AB) S(between runs): 0.000074 Sw(within run): 0.000061 S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges expressed in

grams:  
 80.00045 - 80.00114 for A+B  
 19.99994 - 20.00046 for A-B

## RECOVERIES:

| Number of Data | Expected Conc (mg/L) | Av. Conc Measured (mg/L) | Standard Deviation (1) |
|----------------|----------------------|--------------------------|------------------------|
| 30             | 20000.0              | 20017.1                  | 60.13                  |
| 31             | 2000.0               | 2000.68                  | 14.25                  |

## DUPLICATES:

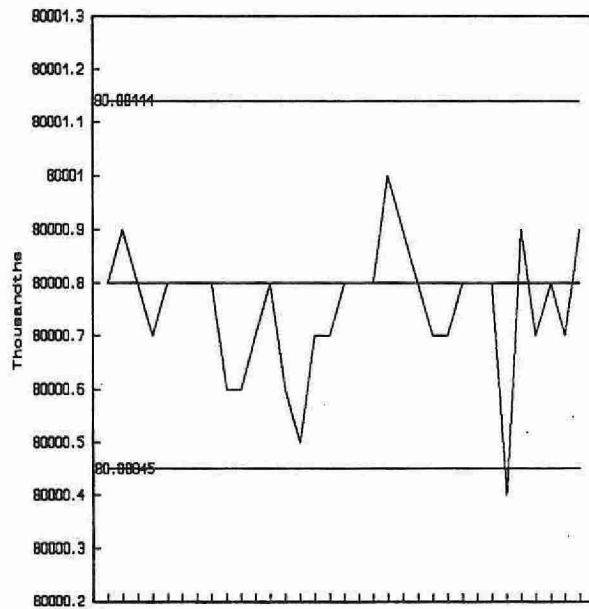
| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|----------------------------------|------------------------|-----------------------------|
| 28           | 0.0 - 100.0                      | 3.4412                 | 7.1                         |
| 10           | 100.1 - 200.0                    | 9.0900                 | 8.5                         |
| 20           | 200.0 - 500.0                    | 6.7627                 | 2.4                         |
| 6            | 500.1 - 2000.0                   | 12.7345                | 1.1                         |
| 64           | OVERALL                          | 6.2774                 |                             |

## OTHER CHECKS:

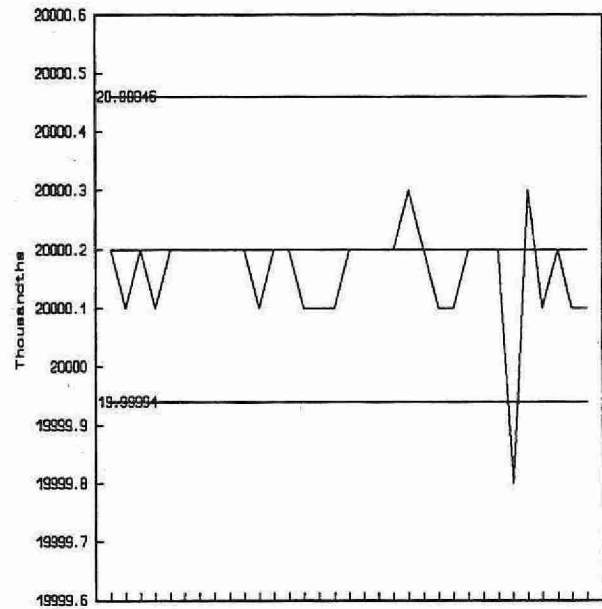
|       | n  | Data Mean (mg/L) | Standard Deviation (1) |
|-------|----|------------------|------------------------|
| Blank | 33 | 2.882            | 8.4500                 |

# **SOLIDS, TOTAL** (mg/L or mg/Kg)

QUALITY CONTROL DATA FROM 14/01/94 TO 30/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

**SOLIDS, TOTAL IGNITED**  
**( Ash and Loss On Ignition)**

**IDENTIFICATION:**

|                      |                          |                   |            |
|----------------------|--------------------------|-------------------|------------|
| Laboratory Unit      | Solids                   | Method Introduced | Before '61 |
| Method Reference No. | E3188B                   | Units             | mg/L       |
| LIMS Product Code    | TIGN3188                 | Supervisor        | J. McBride |
| Sample Type/Matrix   | Sewage, Industrial Waste |                   |            |

**SAMPLING:**

|                   |                  |
|-------------------|------------------|
| Quantity Required | 5-500 mL         |
| Container         | Glass or plastic |

**ANALYTICAL PROCEDURE:**

The procedure for total solids (TS3188) is followed and the dried residue is ignited at 600 50 °C for one hour in a muffle furnace. As soon as practical, the dish is transferred to a desiccator to cool. The ash (fixed solids) is the difference between the final ignited mass plus filter and the original tare weight of the filter, divided by the original sample volume (mL) used for TS3188. The loss on ignition (estimate of volatile total solids) is the difference between the final ignited mass plus filter and the residue (total solids) plus filter, divided by the original sample volume (mL). Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

**INSTRUMENTATION:**

Balance (5 decimal places), muffle furnace, filters, Petri dishes  
Microcomputer system with appropriate software

**REPORTING:**

|                                |                      |                     |
|--------------------------------|----------------------|---------------------|
| Maximum Significant Figures: 3 | Current W value: 2.0 | Current T value: 10 |
|--------------------------------|----------------------|---------------------|

**CONTROLS:**

|             |   |
|-------------|---|
| Calibration | 2 S class weights, e.g. QCA (results in grams)                          |
| Drift       | Balance is reset to zero after every 10 weighings by the microcomputer. |

**NOTES:**

In June 1994, Solids method E3188B was written to include methods for all Solids fractions (Total, Suspended, Dissolved and Ignited) on sewage and industrial waste samples. The former methods which covered these techniques for all matrices, were discontinued ie., E3188A, E3190A, E3192A, and E3194A.

**SOLIDS, TOTAL IGNITED  
(Ash and Loss On Ignition)**

QUALITY CONTROL DATA FROM 01/01/94 TO 31/12/94

Laboratory Unit: Solids

**CALIBRATION CONTROL:**

|      | n  | Expected Mass<br>(g) | Av. Mass<br>Measured (g) | Av.<br>Bias (g) | Standard<br>Deviation (1) |
|------|----|----------------------|--------------------------|-----------------|---------------------------|
| A:   | 32 | 50.00                | 50.00001                 | 0.00001         | 0.00008                   |
| B:   | 32 | 30.00                | 30.00009                 | 0.00009         | 0.00007                   |
| A+B: | 32 | 80.00                | 80.00010                 | 0.00010         | 0.00015                   |
| A-B: | 32 | 20.00                | 19.99992                 | -0.00008        | 0.00004                   |

s.d.(AB)      S(between runs): 0.000077      Sw(within run): 0.000032      S/Sw: 2.4

The calibration is accepted if the calibration control values obtained lie within the ranges expressed in grams:

79.9996      -      80.0004      for      A+B  
19.9997      -      20.0003      for      A-B

**SOLIDS, TOTAL IGNITED ( ASH )**

**DUPLICATES:**

| n<br>Data Pairs | Sample<br>Concentration Span (mg/L) | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|-------------------------------------|---------------------------|--------------------------------|
| 14              | 0 - 2000.0                          | 11.9413                   | 0.8                            |
| 20              | 2000.1 - 5000.0                     | 16.4818                   | 1.3                            |
| 10              | 5000.1 - 10000.0                    | 42.7020                   | 2.3                            |
| 11              | 10000.1 - 25000.0                   | 68.4396                   | 0.4                            |
| 2               | 25000.1 - 50000.0                   | n.a.                      | n.a.                           |
| 57              | OVERALL                             | 28.8311                   |                                |

**OTHER CHECKS:**

|       | n  | Data<br>Mean (mg/L) | Standard<br>Deviation (1) |
|-------|----|---------------------|---------------------------|
| Blank | 32 | 0.805               | 6.3257                    |



**SOLIDS, TOTAL IGNITED**  
(Ash and Loss On Ignition)

**SOLIDS, TOTAL IGNITED ( LOSS ON IGNITION )**

**DUPLICATES:**

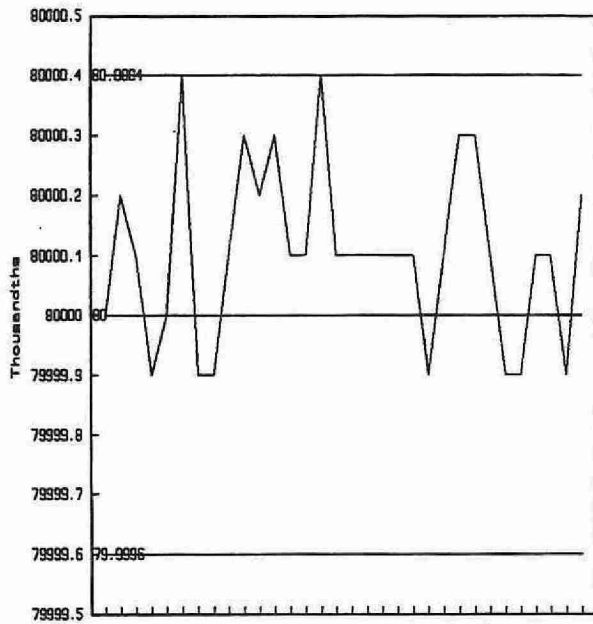
| n<br>Data Pairs | Sample<br>Concentration Span (mg/L) | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|-------------------------------------|---------------------------|--------------------------------|
| 10              | 0 - 2000.0                          | 27.4603                   | 2.0                            |
| 20              | 2000.1 - 5000.0                     | 51.1482                   | 1.9                            |
| 5               | 5000.1 - 10000.0                    | 38.6501                   | 0.4                            |
| 18              | 10000.1 - 25000.0                   | 107.8112                  | 0.7                            |
| 2               | 25000.1 - 50000.0                   | n.a.                      | n.a.                           |
| 55              | OVERALL                             | 58.5900                   | 0.9                            |

**OTHER CHECKS:**

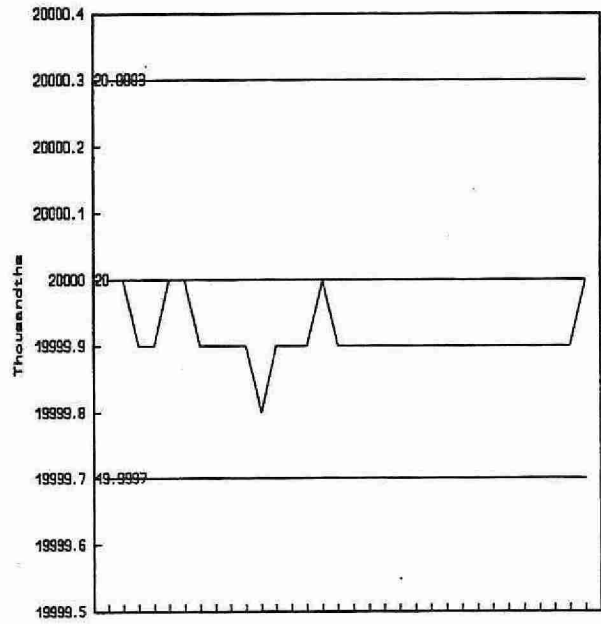
|       | n  | Data<br>Mean (mg/L) | Standard<br>Deviation (1) |
|-------|----|---------------------|---------------------------|
| Blank | 30 | 1.2887              | 6.5828                    |

**SOLIDS, TOTAL IGNITED (mg/L)**  
**(Particulate Ash and Loss on Ignition)**

QUALITY CONTROL DATA FROM 01/01/94 TO 31/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## SULPHATE

### IDENTIFICATION:

|                      |                                      |                   |                         |
|----------------------|--------------------------------------|-------------------|-------------------------|
| Laboratory Unit      | Dorset                               | Method Introduced | 01/04/78                |
| LIS Test Name Code   | SSO4UR                               | Units             | mg/L as SO <sub>4</sub> |
| Work Station Code    | DOIC                                 | Unit Code         | 064941                  |
| Method Code          | 003AI0                               | Supervisor        | J. McBride              |
| Method Reference No. | E3147A                               |                   |                         |
| Sample Type/Matrix   | Precipitation, Throughfall, Stemflow |                   |                         |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 15 mL            |
| Container         | Glass or Plastic |

### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na<sub>2</sub>CO<sub>3</sub>/NaHCO<sub>3</sub> to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO<sub>4</sub> is determined by the comparison of the sample peak heights to a series of standards.

Chloride is determined simultaneously.

### INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |                                  |
|-------------|----------------------------------|
| Calibration | LTBL plus 2 standards, e.g., QCA |
| Drift       | 1 standard every 10 samples.     |

# SULPHATE

QUALITY CONTROL DATA FROM 07/01/94 TO 19/12/94

Laboratory Unit: Dorset

Full Scale: to 10.0 mg/L as SO<sub>4</sub>

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 35 | 8.0                    | 8.003              | 0.003     | 0.1008                 |
| B:   | 35 | 2.0                    | 2.003              | 0.003     | 0.0603                 |
| A+B: | 35 | 10.0                   | 9.985              | -0.015    | 0.1017                 |
| A-B: | 35 | 6.0                    | 6.001              | 0.001     | 0.1235                 |

s.d.(AB) S(between runs): 0.08

Sw(within run): 0.09

S/Sw: 0.95

The calibration is accepted if the calibration control values obtained lie within the ranges:

9.70 - 10.30 for A+B  
5.64 - 6.36 for A-B

## DUPLICATES:

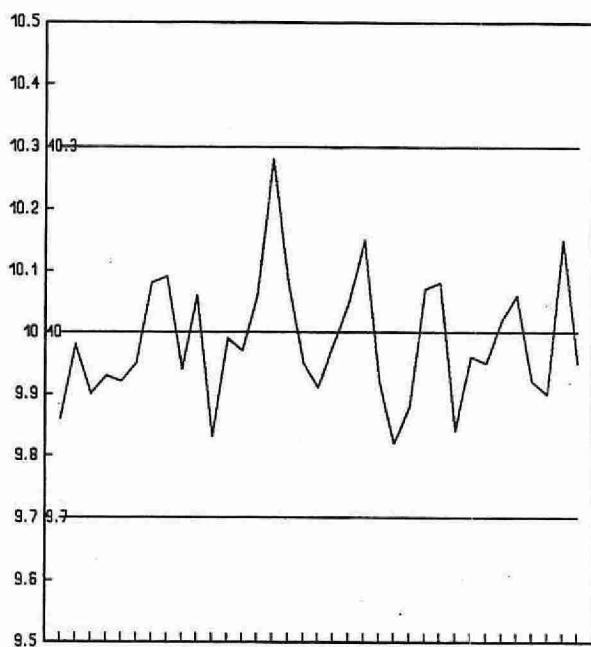
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 14           | 0.00 - 1.00               | 0.0417                 | 20.5                        |
| 12           | 1.01 - 2.00               | 0.0518                 | 3.3                         |
| 32           | 2.01 - 5.00               | 0.0818                 | 2.1                         |
| 69           | 5.01 - 10.0               | 0.0724                 | 1.0                         |
| 127          | Overall                   | 0.0693                 |                             |

## OTHER CHECKS:

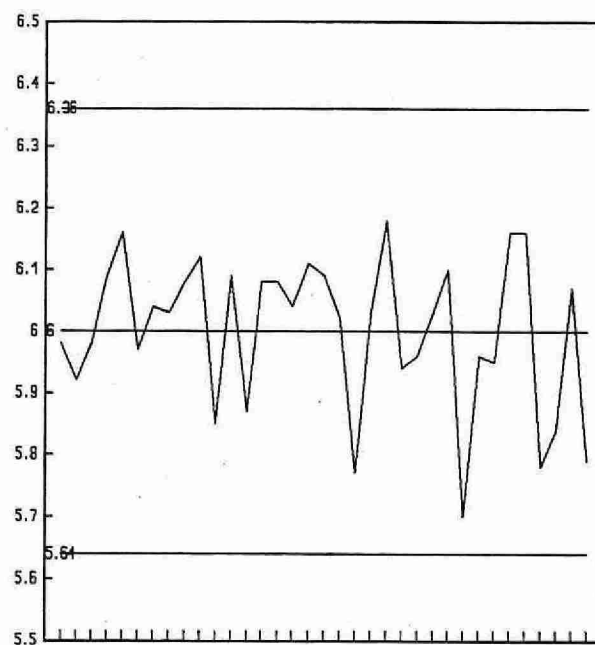
|                 | n  | Mean   | Standard Deviation (1) |
|-----------------|----|--------|------------------------|
| Long Term Blank | 35 | 0.0106 | 0.0400                 |

# SULPHATE (mg/L as SO<sub>4</sub>)

QUALITY CONTROL DATA FROM 07/01/94 TO 19/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## SULPHATE

### IDENTIFICATION:

|                     |  |                   |                                     |
|---------------------|--|-------------------|-------------------------------------|
| Laboratory Unit     | Ion Chromatography   | Method Introduced | 01/04/78                            |
| Method Reference No | E3148A   | Units             | $\mu\text{g}/\text{Filter as SO}_4$ |
| LIMS Product Code   | LOV3148, ANLOV3148,<br>TEF3148, NYL3148,<br>SDIO3148, ANION3148  | Supervisor        | F. Lo                               |
| Sample Type/Matrix  | W40 filters from LoVol filter packs. Teflon filters from Sequential filter packs. Nylon and W41 filters from both LoVol and Sequential filter packs. |                   |                                     |

### SAMPLING:

|                   |   |
|-------------------|---|
| Quantity Required | 1 filter for W40, Teflon or Nylon. 1 set of 2 W41 filters                             |
| Container         | 50 mL polypropylene tube  |
| Other             | For W41 filters, filters are impregnated with potassium carbonate / glycerol solution |

### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW (W40) or 25.0 mL of DDW (Teflon) or 25.0 mL of 0.03 N NaOH (Nylon) in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period. For W41 filters, filters are extracted with 50.0 mL of 0.05%  $\text{H}_2\text{O}_2$  in polypropylene tubes with one hour of mechanical shaking, followed by ultrasonic treatment to enhance extraction, then a 24 hour rest period.  $\text{SO}_2$  is converted to  $\text{SO}_4$  in the process.

### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with  $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$  to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as  $\text{SO}_4$  is determined by the comparison of the sample peak heights to a series of standards. Results are converted to  $\mu\text{g}/\text{filter as SO}_4$  for W40, Teflon and Nylon filters. As for W41 filters, results are converted to  $\mu\text{g}/\text{filter as SO}_2$ . Chloride and nitrogen-nitrate are determined simultaneously.

### INSTRUMENTATION:

Mechanical shaker, Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

### REPORTING:

|                                |                            |                           |
|--------------------------------|----------------------------|---------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.02 mg/L | Current T value: 0.1 mg/L |
|--------------------------------|----------------------------|---------------------------|

### CALIBRATION:

BL plus 9 standards

## SULPHATE cont'd

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift       | 1 standard every 10 samples     |

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received. To convert unit from mg/L to  $\mu\text{g}/\text{Filter}$ , multiply the concentration of  $\text{SO}_4$  in mg/L by 50 for W40 filters or 25 for Teflon or Nylon filters or 33.3 for W41 filters.

# SULPHATE

QUALITY CONTROL DATA FROM 19/01/94 TO 06/12/94

Laboratory Unit: Ion Chromatography

Full Scale: to 10.0 mg/L as SO<sub>4</sub>

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 40 | 8.0                    | 8.010              | 0.010     | 0.0348                 |
| B:   | 40 | 2.0                    | 1.993              | -0.007    | 0.0229                 |
| A+B: | 40 | 10.0                   | 10.003             | 0.003     | 0.0430                 |
| A-B: | 40 | 6.0                    | 6.017              | 0.017     | 0.0040                 |

s.d.(AB) S(between runs): 0.0295

Sw(within run): 0.0285

S/Sw: 1.04

The calibration is accepted if the calibration control values obtained lie within the ranges:

9.68 - 10.32 for A+B  
5.76 - 6.24 for A-B

## DUPLICATES:

For W40 filters:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 4            | 0.00 - 1.00               | 0.0041                 | 0.9                         |
| 5            | 1.01 - 2.00               | 0.0195                 | 1.0                         |
| 22           | 2.01 - 5.00               | 0.0177                 | 0.6                         |
| 6            | 5.01 - 10.00              | 0.0253                 | 0.3                         |
| 37           | Overall                   | 0.0176                 |                             |

For Teflon filters:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 20           | 0.00 - 1.00               | 0.0048                 | 1.0                         |
| 14           | 1.01 - 2.00               | 0.0091                 | 0.6                         |
| 22           | 2.01 - 5.00               | 0.0199                 | 0.5                         |
| 8            | 5.01 - 10.00              | 0.0259                 | 0.4                         |
| 64           | Overall                   | 0.0136                 |                             |



# SULPHATE

QUALITY CONTROL DATA FROM 19/01/94 TO 06/12/94

Laboratory Unit: Ion Chromatography

Full Scale: to 10.0 mg/L as SO<sub>4</sub>

For Nylon filters:

| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 43              | 0.00 - 1.00                  | 0.0127                    | 3.8                            |
| 10              | 1.01 - 2.00                  | 0.0188                    | 1.3                            |
| 20              | 2.01 - 5.00                  | 0.0223                    | 0.7                            |
| 1               | 5.01 - 10.00                 | N.A.                      | N.A.                           |
| 74              | Overall                      | 0.0168                    |                                |

For W41 filters:

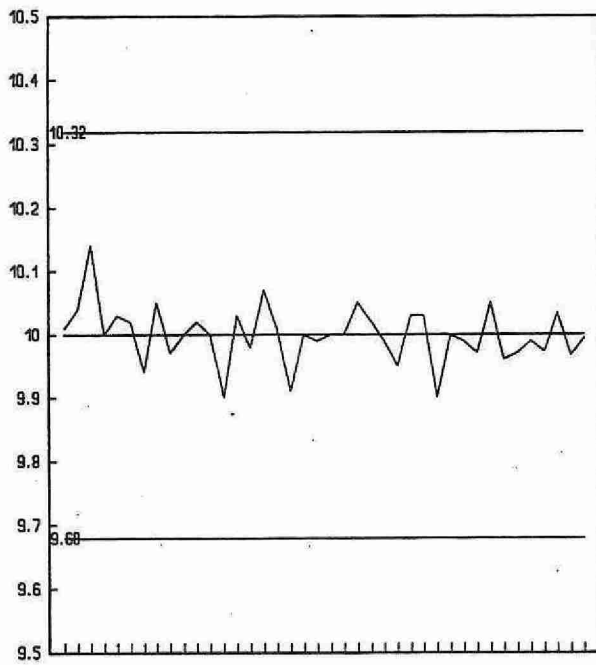
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 31              | 0.00 - 1.00                  | 0.0084                    | 5.1                            |
| 10              | 1.01 - 2.00                  | 0.0233                    | 2.3                            |
| 21              | 2.01 - 5.00                  | 0.0364                    | 1.2                            |
| 10              | 5.01 - 10.00                 | 0.0905                    | 1.4                            |
| 72              | Overall                      | 0.0265                    |                                |

OTHER CHECKS:

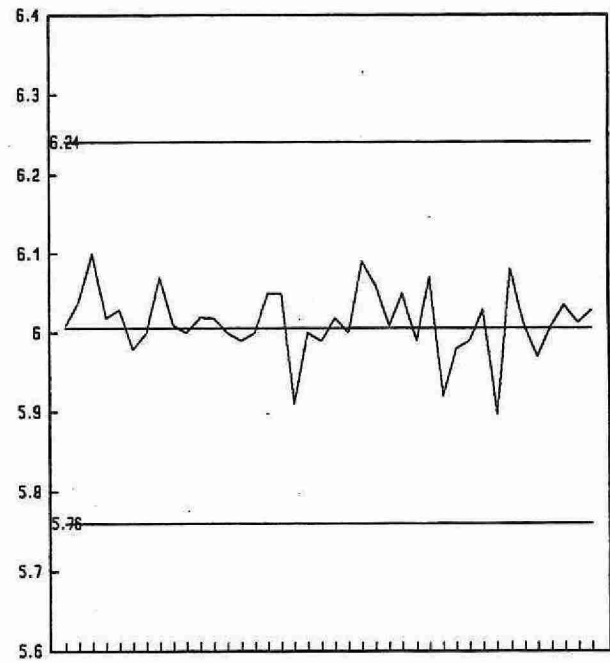
|                 | n  | Mean  | Standard<br>Deviation (1) |
|-----------------|----|-------|---------------------------|
| Long Term Blank | 40 | 0.000 | 0.0000                    |

SULPHATE (mg/L as SO<sub>4</sub>)

QUALITY CONTROL DATA FROM 19/01/94 TO 06/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## SULPHATE

### IDENTIFICATION:

|                     |   |                   |                         |
|---------------------|---|-------------------|-------------------------|
| Laboratory Unit     | Ion Chromatography  | Method Introduced | 01/04/82                |
| Method Reference No | E3172A  | Units             | mg/L as SO <sub>4</sub> |
| LIMS Product Code   | SULP3172  | Supervisor        | F. Lo                   |
| Sample Type/Matrix  | Rivers, Lakes, Domestic Waters, Leachates, Soil Extracts, Effluents |                   |                         |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 50 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the samples by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. The concentration of sulphate in mg/L as SO<sub>4</sub> is determined by comparison of the sample scan to a series of standard scans.

### INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus control module (in-house design) for automated sample introduction and timing.

### REPORTING:

|                                |                      |                      |
|--------------------------------|----------------------|----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |
|--------------------------------|----------------------|----------------------|

### CALIBRATION:

BL plus 10 standards

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift       | 1 standard every 10 samples     |

# SULPHATE

QUALITY CONTROL DATA FROM 04/01/94 TO 21/12/94

Laboratory Unit: Ion Chromatography

Full Scale: to 100.0 mg/L as SO<sub>4</sub>

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 80 | 80.0                   | 80.2               | 0.2       | 0.7239                 |
| B:   | 80 | 20.0                   | 20.1               | 0.1       | 0.5757                 |
| A+B: | 80 | 100.0                  | 100.2              | 0.2       | 1.0207                 |
| A-B: | 80 | 60.0                   | 60.1               | 0.1       | 0.8180                 |

s.d.(AB)      S(between runs): 0.65      Sw(within run): 0.58      S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

97.2      -      102.8      for      A+B  
57.9      -      62.1      for      A-B

## DUPLICATES:

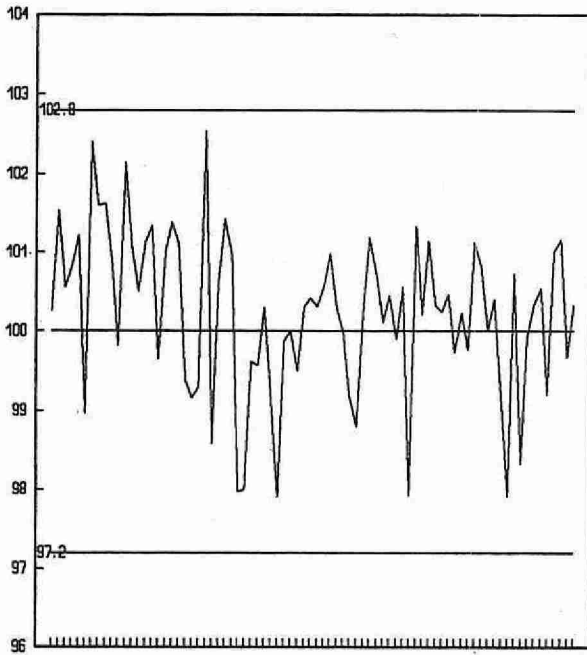
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 47           | 0.0 - 10.0                | 0.1979                 | 6.5                         |
| 44           | 10.1 - 20.0               | 0.4933                 | 3.4                         |
| 93           | 20.1 - 50.0               | 0.7781                 | 2.3                         |
| 24           | 50.1 - 100.0              | 0.9380                 | 1.2                         |
| 208          | Overall                   | 0.6555                 |                             |

## OTHER CHECKS:

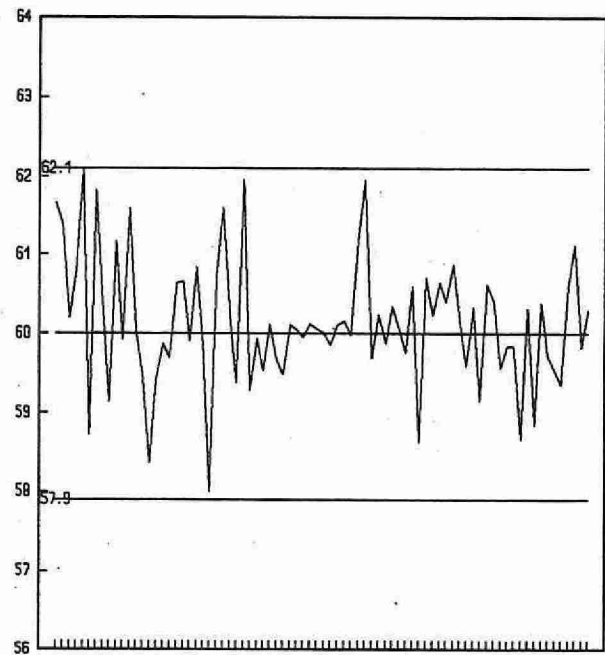
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 80 | 0.445 | 0.1718                 |

SULPHATE (mg/L as SO<sub>4</sub>)

QUALITY CONTROL DATA FROM 04/01/94 TO 21/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## SULPHATE

### IDENTIFICATION:

|                     |                                      |                   |                         |
|---------------------|--------------------------------------|-------------------|-------------------------|
| Laboratory Unit     | Ion Chromatography                   | Method Introduced | 01/04/78                |
| Method Reference No | E3372A                               | Units             | mg/L as SO <sub>4</sub> |
| LIMS Product Code   | ANION3372                            | Supervisor        | F. Lo                   |
| Sample Type/Matrix  | Precipitation, Throughfall, Stemflow |                   |                         |

### SAMPLING:

|                   |                  |
|-------------------|------------------|
| Quantity Required | 15 mL            |
| Container         | Glass or plastic |

### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na<sub>2</sub>CO<sub>3</sub>/NaHCO<sub>3</sub> to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO<sub>4</sub> is determined by the comparison of the sample peak heights to a series of standards. Chloride and nitrogen-nitrate are determined simultaneously.

### INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

|             |                                 |
|-------------|---------------------------------|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift       | 1 standard every 10 samples     |

### NOTES:

Same analytical method as E3147A operating in Dorset Lab. New method number introduced for Toronto Lab in 1993 is E3372A.

# SULPHATE

QUALITY CONTROL DATA FROM 11/01/94 TO 16/12/94

Laboratory Unit: Ion Chromatography

Full Scale: to 5.0 mg/L as SO<sub>4</sub>

## CALIBRATION CONTROL:

|      | n  | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|------|----|------------------------|--------------------|-----------|------------------------|
| A:   | 35 | 4.00                   | 3.993              | -0.007    | 0.0228                 |
| B:   | 35 | 1.00                   | 1.009              | 0.009     | 0.0423                 |
| A+B: | 35 | 5.00                   | 5.002              | 0.002     | 0.0525                 |
| A-B: | 35 | 3.00                   | 2.984              | -0.016    | 0.0432                 |

s.d.(AB) S(between runs): 0.0340 Sw(within run): 0.0305 S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

4.79 - 5.21 for A+B  
2.84 - 3.16 for A-B

## DUPLICATES:

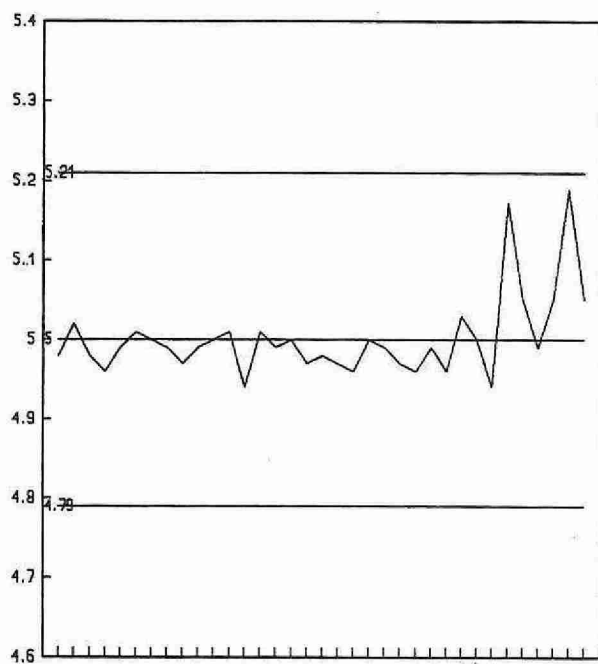
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|--------------|---------------------------|------------------------|-----------------------------|
| 5            | 0.00 - 0.50               | 0.0081                 | 2.0                         |
| 12           | 0.51 - 2.00               | 0.0180                 | 1.4                         |
| 20           | 2.01 - 5.00               | 0.0459                 | 1.4                         |
| 37           | Overall                   | 0.0311                 |                             |

## OTHER CHECKS:

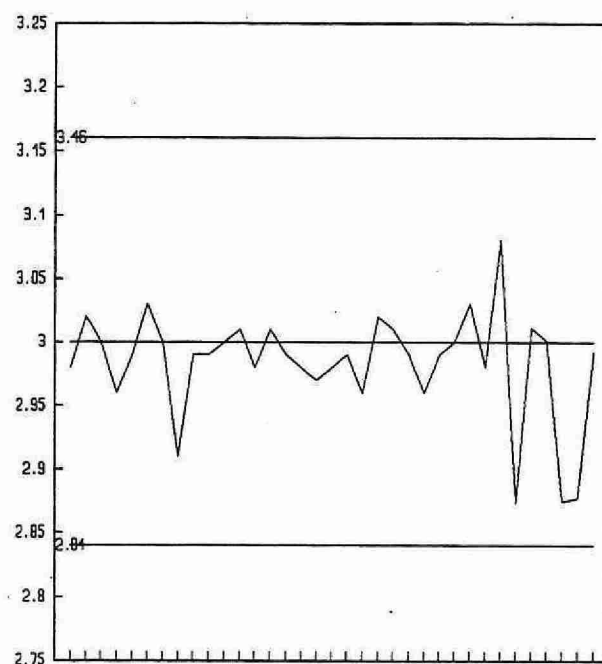
|                 | n  | Mean  | Standard Deviation (1) |
|-----------------|----|-------|------------------------|
| Long Term Blank | 35 | 0.000 | 0.0000                 |

# SULPHATE (mg/L as SO<sub>4</sub>)

QUALITY CONTROL DATA FROM 11/01/94 TO 16/12/94



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT



## TURBIDITY

### IDENTIFICATION:

|                      |  |                    |             |
|----------------------|--|--------------------|-------------|
| Laboratory Unit:     | Colourimetry   | Method Introduced: | Before '74  |
| Method Reference No: | E3311A   | Units:             | FTU         |
| LIMS Product Code:   | TURB3311   | Supervisor:        | M. Rawlings |
| Sample Type/Matrix:  | Rivers, Lakes, Effluents, Drinking Water, Industrial Waste, Sewage |                    |             |

### SAMPLING:

|                    |                  |
|--------------------|------------------|
| Quantity Required: | 50 mL            |
| Container:         | Glass or plastic |

### ANALYTICAL PROCEDURE:

The instrument is standardized with sealed standards which are prepared commercially and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

### INSTRUMENTATION:

-Hach Ratio/XR Model Turbidimeter modified to accept control signals from robot controller, electronic interphase, Zymark ZYMATE 11 Laboratory Robot System, IBM PC computer.

### REPORTING:

|                                |                       |                       |
|--------------------------------|-----------------------|-----------------------|
| Maximum Significant Figures: 3 | Current W value: 0.01 | Current T value: 0.05 |
|--------------------------------|-----------------------|-----------------------|

### CALIBRATION:

BL plus formazin standards (once every four months)

### CONTROLS:

|              |                       |
|--------------|-----------------------|
| Calibration: | 5 standards, e.g. QCA |
|--------------|-----------------------|

### NOTES:

The QCC outlier in January was caused by incorrect alignment of the Gelex standard in the turbidimeter. No sample results were reported in the QCC range (20 - 200 FTU).  
In October the outlier were due to insufficient warm up time of the turbidimeter.

# TURBIDITY

QUALITY CONTROL DATA FROM 06/01/94 TO 23/12/94

Laboratory Unit: Colourimetry

Full Scale: to 2000 FTU

**CALIBRATION CONTROL: FROM 06/01/94 TO 31/05/94**

|    | n  | Expected<br>Concentration | Mean<br>Concentration | Standard<br>Deviation (1) |
|----|----|---------------------------|-----------------------|---------------------------|
| A: | 71 | 2.0                       | 1.06                  | 0.0126                    |
| B: | 71 | 20.0                      | 15.99                 | 0.1120                    |
| C: | 71 | 200.0                     | 166.30                | 1.3118                    |
| D: | 71 | 2000.0                    | 1225.31               | 6.0512                    |

On any given day the calibration is accepted if the values obtained lie within the ranges:

|       |   |       |       |
|-------|---|-------|-------|
| 0.891 | - | 1.223 | for A |
| 15.46 | - | 16.4  | for B |
| 161.8 | - | 170.8 | for C |
| 1204  | - | 1244  | for D |

**OTHER CHECKS:**

|             | n  | Data<br>Mean | Standard<br>Deviation (1) |
|-------------|----|--------------|---------------------------|
| Stray Light | 71 | 0.0444       | 0.0016                    |

**CALIBRATION CONTROL: FROM 01/06/94 TO 23/12/94 (for A,B,C)      FROM 01/06/94 TO 26/08/94 (for D)**

|    | n   | Expected<br>Concentration | Mean<br>Concentration | Standard<br>Deviation (1) |
|----|-----|---------------------------|-----------------------|---------------------------|
| A: | 103 | 2.0                       | 1.189                 | 0.0519                    |
| B: | 103 | 20.0                      | 13.967                | 0.1097                    |
| C: | 103 | 200.0                     | 173.494               | 1.2403                    |
| D: | 47  | 2000.0                    | 1367.979              | 5.9727                    |

On any given day the calibration is accepted if the values obtained lie within the ranges:

|       |   |       |       |
|-------|---|-------|-------|
| 1.007 | - | 1.339 | for A |
| 13.22 | - | 14.16 | for B |
| 166.7 | - | 175.7 | for C |
| 1357  | - | 1397  | for D |

**OTHER CHECKS:**

|             | n   | Data<br>Mean | Standard<br>Deviation (1) |
|-------------|-----|--------------|---------------------------|
| Stray Light | 103 | 0.0488       | 0.0018                    |

**CALIBRATION CONTROL: FROM 31/08/94 TO 23/12/94**

|    | n  | Expected<br>Concentration | Mean<br>Concentration | Standard<br>Deviation (1) |
|----|----|---------------------------|-----------------------|---------------------------|
| D: | 56 | 2000.0                    | 1426.107              | 6.2542                    |

On any given day the calibration is accepted if the values obtained lie within the ranges:

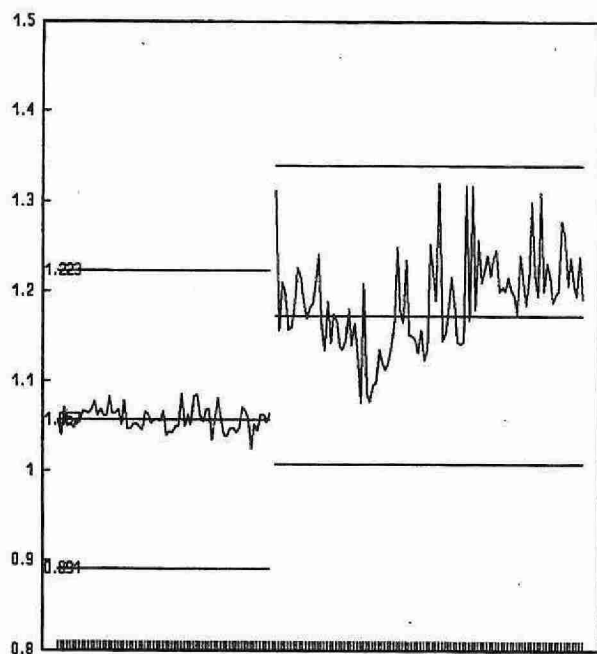
1407 - 1447 for D

**DUPLICATES:**

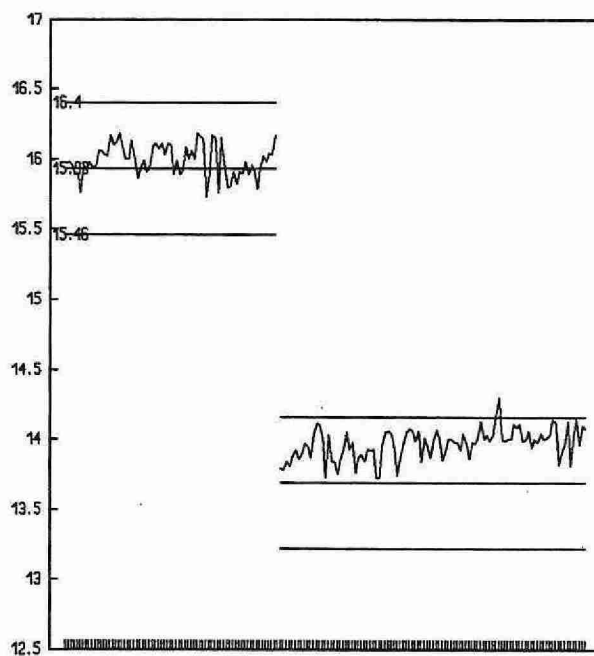
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 256             | 0.0 - 2.0                    | 0.0519                    | 13.9                           |
| 160             | 2.0 - 20.0                   | 0.5728                    | 14.0                           |
| 38              | 20.0 - 200.0                 | 2.3772                    | 6.6                            |
| 7               | 200.0 - 2000.0               | 9.2800                    | 1.6                            |
| 461             | Overall                      | 0.2645                    |                                |

# TURBIDITY (FTU)

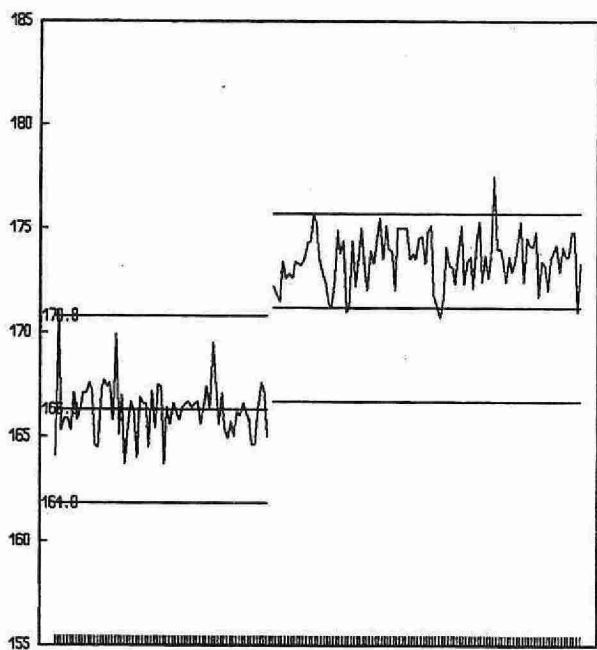
QUALITY CONTROL DATA FROM 06/01/94 TO 23/12/94



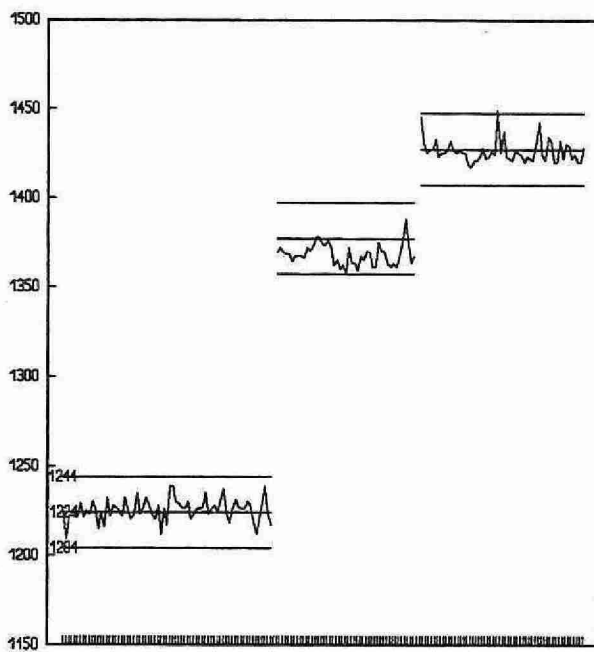
QUALITY CONTROL STANDARD A



QUALITY CONTROL STANDARD B



QUALITY CONTROL STANDARD C



QUALITY CONTROL STANDARD D

CONTROL LIMIT

## ZINC, TOTAL

### IDENTIFICATION:

|                      |                               |                   |            |
|----------------------|-------------------------------|-------------------|------------|
| Laboratory Unit      | Dorset                        | Method Introduced | 1991       |
| LIS Test Name Code   | ZNUT                          | Units             | µg/L as Zn |
| Work Station Code    | DOTRACE                       | Unit Code         | 063830     |
| Method Code          | 005AF2                        | Supervisor        | J. McBride |
| Method Reference No. | E3376A                        |                   |            |
| Sample Type/Matrix   | Surface waters, precipitation |                   |            |

### SAMPLING:

|                   |  |
|-------------------|--|
| Quantity Required | 5 mL   |
| Container         | Glass or plastic, capped, acidified to 0.25% with HNO <sub>3</sub> |

### ANALYTICAL PROCEDURE:

Samples are analyzed by GFAAS at 213.9 nm.  
Approximate absorbance: 0.8 at full scale level

### INSTRUMENTATION:

Varian graphite furnace atomic absorption spectrometer with automated sampler.

### REPORTING:

|                                |                        |                        |
|--------------------------------|------------------------|------------------------|
| Maximum Significant Figures: 3 | Current W value: 0.001 | Current T value: 0.005 |
|--------------------------------|------------------------|------------------------|

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

|             |                             |
|-------------|-----------------------------|
| Calibration | 1 NRC sample, 3 duplicates  |
| Drift       | 1 standard every 10 samples |

# ZINC, TOTAL

QUALITY CONTROL DATA FROM 04/04/94 TO 22/12/94

Laboratory Unit: Dorset

Full Scale: to 20 µg/L as Zn

## CALIBRATION CONTROL:

|      | n  | Mean<br>Concentration | Standard<br>Deviation (1) |
|------|----|-----------------------|---------------------------|
| QCA: | 25 | 2.60                  | 0.0902                    |
| NRC: | 25 | 3.35                  | 0.0795                    |

## DUPLICATES:

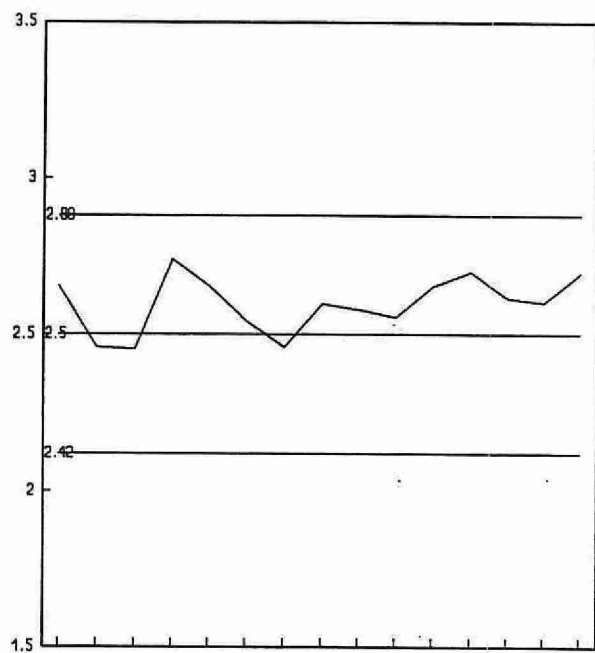
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|-----------------|------------------------------|---------------------------|--------------------------------|
| 18              | 0.0 - 2.0                    | 0.2209                    | 18.9                           |
| 16              | 2.1 - 4.0                    | 0.3732                    | 19.4                           |
| 11              | 4.1 - 10.0                   | 0.2496                    | 5.0                            |
| 0               | 10.1 - 20.0                  | N.A.                      | N.A.                           |
| 45              | Overall                      | 0.2808                    |                                |

## OTHER CHECKS:

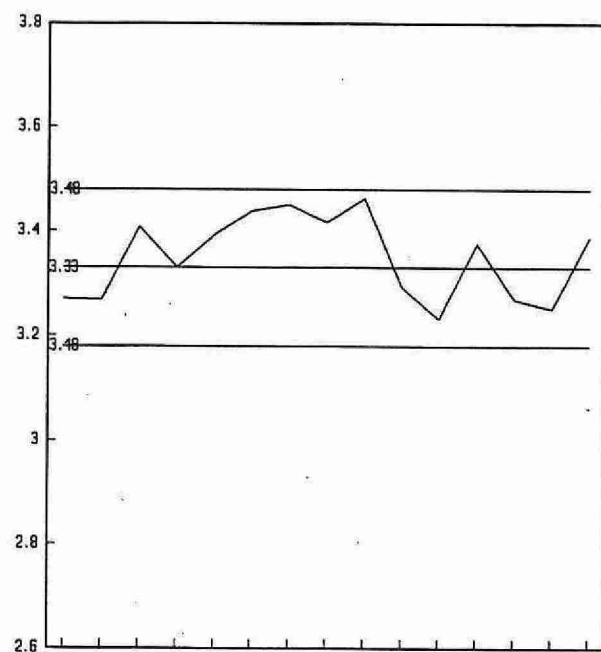
|     | n  | Mean   | Standard<br>Deviation (1) |
|-----|----|--------|---------------------------|
| NRC | 15 | 3.3499 | 0.0796                    |

# ZINC, TOTAL (µg/L)

QUALITY CONTROL DATA FROM 04/04/94 TO 22/12/94



QUALITY CONTROL STANDARD A



NRC REFERENCE SAMPLE

CONTROL LIMIT

## BIBLIOGRAPHY

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4. Laboratory Services Branch, Water Quality Section, *Performance Report*. S. Janhurst. 1993.
5. Laboratory Service Branch, Water Quality Section, *Standard Operating Procedure for Method Intercomparison*. M. Rawlings. December 1990.
6. Laboratory Service Branch, Water Quality Section, *Regression Techniques for Analytical Chemistry Technicians*. M. Rawlings. January 1991.
7. Laboratory Services Branch, *A Guide to the Collection and Submission of Samples for Laboratory Analysis*. 1993.
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## ABBREVIATIONS

|         |   |
|---------|---|
| AAS     | - Atomic Absorption Spectrophotometer                             |
| Abs     | - Absorbance  |
| APIOS   | - Acidic Precipitation in Ontario Study                           |
| Av      | - Average   |
| Bl      | - Blank   |
| C       | - Degree Centigrade   |
| cm      | - Centimetre  |
| Concn   | - Concentration   |
| Date    | - Day/Month/Year  |
| DDW     | - Deionized distilled water                                       |
| Pure-DW | - Pure Deionized Water  |
| DO      | - Dissolved oxygen  |
| DW      | - Distilled water   |
| ECSS    | - Expert Committee on Soil Survey (Land Resource Research Centre) |
| EPA     | - Environmental Protection Agency                                 |
| FTU     | - Formazin Turbidity Units  |
| g       | - Gram  |
| HOAC    | - Acetic Acid   |
| HZU     | - Hazen Units   |
| IR      | - Infra-Read  |
| L       | - Litre   |
| LAB     | - Laboratory  |
| LIMS    | - Laboratory Information Management System                        |
| LIS     | - Laboratory Information System                                   |
| LTBL    | - Long Term Blank   |
| M       | - Molar   |
| meq     | - Milliequivalent   |
| mg      | - Milligram   |
| min     | - Minute  |

## ABBREVIATIONS cont'd

|                |  |
|----------------|--|
| mL             | - Millilitre   |
| mm             | - Millimetre   |
| N/A            | - Not Available or Not Applicable                      |
| nm             | - Nanometre  |
| NRC            | - National Research Council                            |
| QC             | - Quality Control                                      |
| QCA            | - Quality Control Standard A                           |
| QCB            | - Quality Control Standard B                           |
| QCC            | - Quality Control Standard C                           |
| QCD            | - Quality Control Standard D                           |
| R              | - Recovery   |
| rpm            | - Revolutions per minute                               |
| S              | - Between run standard deviation                       |
| S <sub>1</sub> | - Standard Deviation (Conventional)                    |
| S <sub>2</sub> | - Standard Deviation for duplicates                    |
| S <sub>w</sub> | - Within run standard deviation                        |
| S. Class       | - Weight classification designation (not certified)    |
| s.d.           | - Standard deviation                                   |
| Standard Cal   | - Colourimeter setting to control electronic expansion |
| STD            | - Standard   |
| TCU            | - True Colour Units                                    |
| µm             | - Micrometer   |
| µeq            | - Microequivalent                                      |
| µg             | - Microgram  |
| µS             | - Micro-Siemen   |
| UV             | - Ultra-Violet   |
| V/V            | - Concentration based on volume measurements           |

## APPENDIX A

### W & T :

W and T are low level data qualifiers assigned to data that are near or below the detection limit values (3)(8). The code <W indicates that no measurable response was observed under the test conditions. The reported value indicates the minimum amount of analyte that could have been measured under routine conditions. W is usually less than the standard deviation of duplicates near zero. The <T code is used to represent a measurable amount of the analyte which under the test conditions is not verifiable. The reported result should be used only for large batches of similar data to evaluate background levels or trends of contaminants in the environment where more sensitive analytical methods are not available.

To provide a consistent Laboratory Services Branch approach to data reporting, the Water Quality Analyses Section calculates W from the standard deviation of duplicates ( $S_2$ ), near zero, by rounding down to the nearest 1, 2 or 5 digit. T is five times W. W and T values for this report are summarized in Appendix B.

## APPENDIX B

## W AND T VALUES FOR DATA REPORTED IN 1994

| PARAMETER                     | UNITS                   | METHOD<br>REFERENCE NO. | FULL<br>SCALE | W     | T     |
|-------------------------------|-------------------------|-------------------------|---------------|-------|-------|
| Acidity, Gran                 | µg/L H <sup>+</sup>     | E3248A                  | 1000          | 1.0   | 5.0   |
| Acidity, Total Fixed Endpoint | mg/L CaCO <sub>3</sub>  | E3248A                  | 100           | 0.05  | 0.25  |
| Alkalinity,<br>Gran           | mg/L CaCO <sub>3</sub>  | E3042A                  | 25            |       |       |
|                               | mg/L CaCO <sub>3</sub>  | E3289A                  | 25            |       |       |
| Total Fixed Endpoint 4.5      | mg/L CaCO <sub>3</sub>  | E3042A                  | 100           | 0.05  | 0.25  |
|                               | mg/L CaCO <sub>3</sub>  | E3218A                  | 1000          | 0.2   | 1.0   |
|                               | mg/L CaCO <sub>3</sub>  | E3228A                  | 1000          | 0.5   | 2.5   |
|                               | mg/L CaCO <sub>3</sub>  | E3289A                  | 1000          | 0.2   | 1.0   |
| Total Fixed Endpoint 3.8      | mg/L CaCO <sub>3</sub>  | E3042A                  | 100           | 0.05  | 0.25  |
| Aluminum,<br>Reactive Species | µg/L as Al              | E3020A                  | 1000          | 2     | 10    |
| Reactive Species              | µg/L as Al              | E3256A                  | 1000          | 2     | 10    |
| Total                         | µg/L as Al              | E3300A                  | 1000          | 2     | 10    |
| Cadmium, Total                | µg/L as Cd              | E3376A                  | 5             | 0.001 | 0.005 |
| Calcium,                      | mg/L as Ca              | E3146A                  | 2             | 0.005 | 0.025 |
|                               | mg/L as Ca              | E3171A                  | 40            | 0.05  | 0.25  |
|                               | mg/L as Ca              | E3217A                  | 200           | 0.2   | 1     |
|                               | mg/L as Ca              | E3249A                  | 8             | 0.02  | 0.1   |
| Carbon, Dissolved Inorganic   | mg/L as C               | E3028A                  | 10            | 0.02  | 0.1   |
|                               | mg/L as C               | E3370A                  | 40            | 0.2   | 1     |
|                               | mg/L as C               | E3370A                  | 80            | 0.2   | 1     |
| Carbon, Dissolved Organic     | mg/L as C               | E3370A                  | 20            | 0.1   | 0.5   |
| Carbon, Total Organic         | mg/L as C               | E3247B                  | 25            | 0.2   | 1     |
| Chloride                      | mg/L as Cl              | E3016A                  | 100           | 0.2   | 1     |
|                               | mg/L as Cl              | E3147A                  | 2             | 0.01  | 0.05  |
|                               | µg/filt as Cl           | E3148A                  | 100           | 1     | 5     |
|                               | mg/L as Cl              | E3372A                  | 1             | 0.01  | 0.05  |
| Chlorine, Total Residual      | µg/L as Cl <sub>2</sub> | E3309A                  | 50            | 2     | 10    |
| Chlorophyll "a"               | µg/L                    | E3169A                  | 50            | 0.2   | 1     |
| Chlorophyll "a" acidified     | µg/L                    | E3169A                  | 10            | 1     | 5     |
| Chlorophyll "b"               | µg/L                    | E3169A                  | 10            | 0.1   | 0.5   |
| Colour, True                  | TCU                     | E3025A                  | 100           | 0.2   | 1     |
|                               | TCU                     | E3219A                  | 100           | 0.2   | 1     |

## APPENDIX B

## W AND T VALUES FOR DATA REPORTED IN 1994

| PARAMETER                          | UNITS                                   | METHOD<br>REFERENCE NO. | FULL<br>SCALE | W     | T     |
|------------------------------------|---|-------------------------|---------------|-------|-------|
| Conductivity                       | $\mu\text{S}/\text{cm}$                 | E3024B                  | 500           | 0.2   | 1     |
|                                    | $\mu\text{S}/\text{cm}$                 | E3177A                  | 100           | 0.2   | 1     |
|                                    | $\mu\text{S}/\text{cm}$                 | E3218A                  | 2000          | 1     | 5     |
|                                    | $\mu\text{S}/\text{cm}$                 | E3228A                  | 10000         | 5     | 25    |
|                                    | $\mu\text{S}/\text{cm}$                 | E3289A                  | 2000          | 1     | 5     |
| Copper,<br>Total                   | $\mu\text{g}/\text{L}$ as Cu            | E3376A                  | 10            | 0.003 | 0.015 |
| Cyanide,<br>Free                   | $\text{mg}/\text{L}$ as Cn              | E3014A                  | 0.2           | 0.001 | 0.005 |
| Total                              | $\text{mg}/\text{L}$ as Cn              | E3015A                  | 0.2           | 0.001 | 0.005 |
| Fluoride                           | $\mu\text{g}/\text{L}$ as F             | E3041A                  | 70            | 0.2   | 1.0   |
|                                    | $\text{mg}/\text{L}$ as F               | E3369A                  | 2             | 0.01  | 0.05  |
| Hardness                           | $\text{mg}/\text{L}$ as $\text{CaCO}_3$ | E3171A                  |               | 0.2   | 1.0   |
|                                    | $\text{mg}/\text{L}$ as $\text{CaCO}_3$ | E3217A                  |               | 0.5   | 2.5   |
|                                    | $\text{mg}/\text{L}$ as $\text{CaCO}_3$ | E3249A                  |               | 0.05  | 0.25  |
| Iron,<br>Total                     | $\mu\text{g}/\text{L}$ as Fe            | E3303B                  | 1000          | 2     | 10    |
| Lead,<br>Total                     | $\mu\text{g}/\text{L}$ as Pb            | E3376A                  | 10            | 0.003 | 0.015 |
| Magnesium                          | $\text{mg}/\text{L}$ as Mg              | E3146A                  | 0.5           | 0.001 | 0.005 |
|                                    | $\text{mg}/\text{L}$ as Mg              | E3171A                  | 10            | 0.02  | 0.1   |
|                                    | $\text{mg}/\text{L}$ as Mg              | E3217A                  | 50            | 0.05  | 0.25  |
|                                    | $\text{mg}/\text{L}$ as Mg              | E3249A                  | 2             | 0.005 | 0.025 |
| Manganese, Total                   | $\mu\text{g}/\text{L}$                  | E3303B                  | 200           | 1     | 5     |
| Nitrogen,<br>Ammonia plus Ammonium | $\text{mg}/\text{L}$ as N               | E3149A                  | 2             | 0.002 | 0.01  |
|                                    | $\mu\text{g}/\text{filt}$ as N          | E3149A                  | 50            | 0.05  | 0.25  |
|                                    | $\text{mg}/\text{L}$ as N               | E3364A                  | 2             | 0.002 | 0.01  |
|                                    | $\text{mg}/\text{L}$ as N               | E3366A                  | 50            | 0.05  | 0.25  |
|                                    | $\mu\text{g}/\text{L}$ as N             | E3374A                  | 1000          | 1     | 5     |
| Nitrogen, Nitrate                  | $\mu\text{g}/\text{filt}$ as N          | E3148A                  | (W40) 100     | 0.5   | 2.5   |
|                                    | $\mu\text{g}/\text{filt}$ as N          | E3148A                  | (Nylon) 50    | 0.2   | 1     |
|                                    | $\mu\text{g}/\text{filt}$ as N          | E3148A                  | (Teflon) 50   | 0.2   | 1     |
|                                    | $\text{mg}/\text{L}$ as N               | E3372A                  | 1             | 0.01  | 0.05  |

## APPENDIX B

## W AND T VALUES FOR DATA REPORTED IN 1994

| PARAMETER                               | UNITS        | METHOD<br>REFERENCE NO. | FULL<br>SCALE | W      | T      |
|---|--------------|-------------------------|---------------|--------|--------|
| Nitrogen, Nitrate plus Nitrite          | µg/L as N    | E3374                   | 1000          | 2.0    | 10     |
|   | mg/L as N    | E3364A                  | 5             | 0.005  | 0.025  |
|   | mg/L as N    | E3366A                  | 50            | 0.05   | 0.25   |
|   | mg/L as N    | E3369A                  | 20            | 0.1    | 0.5    |
| Nitrogen, Nitrite                       | mg/L as N    | E3364A                  | 0.2           | 0.001  | 0.005  |
|   | mg/L as N    | E3366A                  | 2             | 0.005  | 0.025  |
| Nitrogen, Total Kjeldahl                | mg/g as N    | E3116A                  | (Sediment) 20 | 0.05   | 0.25   |
|   | mg/g as N    | E3118A                  | (Soil) 20     | 0.1    | 0.5    |
|   | mg/L as N    | E3367A                  | 2             | 0.02   | 0.1    |
|   | mg/L as N    | E3368A                  | 50            | 0.05   | 0.25   |
| Oxygen Demand, Biochemical              | mg/L as O    | E3182A                  | 9             | 0.2    | 1.0    |
| Oxygen Demand, Chemical                 | mg/L as O    | E3170A                  | 40            | 1.0    | 5.0    |
|   | mg/L as O    | E3246A                  | 500           | 2.0    | 10     |
| pH                                      |              | E3042A                  | 14            |        |        |
|   |              | E3218A                  | 14            |        |        |
|   |              | E3228A                  | 14            |        |        |
|   |              | E3248A                  | 14            |        |        |
|   |              | E3289A                  | 14            |        |        |
| Phenolics, Reactive                     | µg/L Phenol  | E3179A                  | 50            | 0.2    | 1.0    |
| Phosphorus,<br>Reactive ortho-Phosphate | mg/L as P    | E3364A                  | 0.1           | 0.0005 | 0.0025 |
|   | mg/L as P    | E3366A                  | 10            | 0.02   | 0.1    |
| Phosphorus, Total                       | µg/L as P    | E3036A                  | 100           | 0.2    | 1.0    |
|   | mg/g as P    | E3116A                  | (Sediment) 5  | 0.01   | 0.05   |
|   | mg/g as P    | E3116A                  | (Soil) 5      | 0.02   | 0.1    |
|   | mg/g as P    | E3118A                  | 8             | 0.02   | 0.1    |
|   | mg/L as P    | E3367A                  | 0.2           | 0.002  | 0.01   |
|   | mg/L as P    | E3368A                  | 10            | 0.02   | 0.1    |
|   | mg/L as P    | E3036A                  | 100           | 0.2    | 1      |
| Potassium,                              | µg/filt as K | E3146A                  | 50            | 0.1    | 0.5    |
|   | mg/L as K    | E3146A                  | 1             | 0.002  | 0.010  |
|   | mg/L as K    | E3171A                  | 5             | 0.01   | 0.05   |
|   | mg/L as K    | E3217A                  | 25            | 0.05   | 0.25   |
|   | mg/L as K    | E3249A                  | 1             | 0.005  | 0.025  |
| Silicon, Reactive Silicates             | mg/L as Si   | E3370A                  | 10            | 0.02   | 0.1    |

# APPENDIX B

## W AND T VALUES FOR DATA REPORTED IN 1994

| PARAMETER                            | UNITS                      | METHOD<br>REFERENCE NO. | FULL<br>SCALE | W     | T     |
|--------------------------------------|----------------------------|-------------------------|---------------|-------|-------|
| Sodium                               | µg/filt as Na              | E3146A                  | 50            | 0.1   | 0.5   |
|                                      | mg/L as Na                 | E3146A                  | 1             | 0.002 | 0.01  |
|                                      | mg/L as Na                 | E3171A                  | 20            | 0.02  | 0.1   |
|                                      | mg/L as Na                 | E3217A                  | 100           | 0.2   | 1     |
|                                      | mg/L as Na                 | E3249A                  | 4             | 0.005 | 0.025 |
| Solids, Dissolved                    | mg/L                       | E3188B                  | 5000          | 2     | 10    |
|                                      | mg/L                       | E3365A                  | 5000          | 2     | 10    |
| Solids, Suspended                    | mg/L                       | E3188B                  | 30000         | 0.5   | 2.5   |
|                                      | mg/L                       | E3365A                  | 5000          | 0.5   | 2.5   |
| Ignited (P. Ash/P. Loss on Ignition) | mg/L                       | E3188B                  | 5000          | 0.5   | 2.5   |
| Solids, Total                        | mg/L or mg/Kg              | E3188B                  | 60000         | 2     | 10    |
|                                      | mg/L or mg/Kg              | E3365A                  | 2000          | 2     | 10    |
| Ignited (Ash and Loss on Ignition)   | mg/L                       | E3188B                  | 50000         | 2     | 10    |
| Sulphate                             | mg/L as SO <sub>4</sub>    | E3147A                  | 10            | 0.05  | 0.25  |
|                                      | µg/filt as SO <sub>4</sub> | E3148A                  | 500           | 1.0   | 5.0   |
|                                      | µg/filt as SO <sub>4</sub> | E3148A                  | (W40) 250     | 1.0   | 5.0   |
|                                      | µg/filt as SO <sub>4</sub> | E3148A                  | (Nylon) 250   | 1.0   | 5.0   |
|                                      | µg/filt as SO <sub>4</sub> | E3148A                  | (Teflon) 250  | 1.0   | 5.0   |
|                                      | mg/L as SO <sub>4</sub>    | E3172A                  | 100           | 0.5   | 2.5   |
|                                      | mg/L as SO <sub>4</sub>    | E3372A                  | 5             | 0.05  | 0.25  |
| Sulphur Dioxide                      | µg/filt as SO <sub>2</sub> | E3148A                  | 350           | 1.0   | 5.0   |
| Turbidity                            | FTU                        | E3311A                  | 2000          | 0.01  | 0.05  |
| Zinc,                                |                            |                         |               |       |       |
| Total                                | µg/L as Zn                 | E3304A                  | 20            | 0.001 | 0.005 |



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